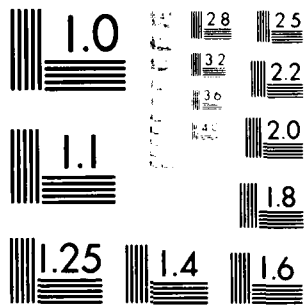


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REPORT NO. QAR-R-004

STATISTICAL GUIDE FOR DETERMINING THE EQUIVALENCY OF TWO CHEMICAL TEST METHODS

ERIC BIXON

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STATISTICAL GUIDE FOR DETERMINING THE EQUIVALENCY
OF TWO CHEMICAL TEST METHODS

Eric R. Bixon

Report No. QAR-R-004

April 1982

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ABSTRACT

This paper is to be used as a guide in gathering and treating data to be used in comparing two chemical test methods to determine equivalency. The concept of an equivalent test method is well defined in terms of repeatability and reproducibility as well as accuracy and precision. All statistical tests and calculations are explained and illustrated using numerical examples.

It should be noted that this is a preliminary report subject to modification after finalized reviews of interested parties.



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INTRODUCTION

DETERMINATION OF THE EQUIVALENCY OF TWO CHEMICAL TEST METHODS

This paper is designed as a statistical guide for determining the equivalency of two chemical test methods. A variety of tests are performed on the data which is obtained by two methods:

a. Method 1 is the specified method that has been referred to in the specification.

b. Method 2 is the proposed method which is being compared to the specified method to determine if it is equivalent or not.

The term equivalency must be defined for this paper. In general the equivalency of two test methods implies that the methods have the same accuracy and the same precision. This is the only case in which the two test methods may be deemed equivalent. If method 1 is more or less accurate or precise than method 2, then the methods are not equivalent.

This guide is divided into four parts. The first two parts represent data which must be gathered and treated by the proponent of the new test method. The results obtained in this section will provide guidance as to whether a round robin should even be performed. If the results obtained in the first two parts indicate that the proposed test method may be an equivalent method, then the round robin should be organized and the data obtained for treatment in Parts 3 and 4.

The data used in Parts 3 and 4 is essentially data from five labs done on three samples. This data is used for evaluating the repeatability and reproducibility associated with each method.

In statistical terms the two methods may be considered equivalent if they have equal means and variances. That is, the methods are equivalent if we can demonstrate that the sample means and variances generated by each method are from the same population. Another criteria is that the lab to lab variation be the same for both methods.

In conjunction with the determination of the equivalency of both methods, this paper illustrates the determination of each methods repeatability and reproducibility using analysis of variance (ANOVA). The conditions necessary to perform ANOVA are stated and the calculations necessary to perform a one-way ANOVA and a two

way ANOVA are illustrated. After these ANOVAs have been determined, the method of using the components of variance to estimate the method repeatability and reproducibility is shown. It should be noted that this paper uses the method of ANOVA to characterize the variability of each method. It does not attempt to differentiate between one lab and the next.

It should be emphasized that this paper is basically an instructional guide to indicate the various tests and operations that can be performed on the data to determine equivalency. The data used in this paper is ideal since it has been fashioned as an illustrative example. In each case the actual experimental data obtained using each method must be examined to see if all necessary conditions are fulfilled in order that the approaches used in this paper can be utilized.

Computer programs have been generated which facilitate all of the time consuming calculations which are required for ANOVA. Data generated from these round robins should be submitted to DRDAR-QAR-R. Sample data sheets, letters for round robin participants, and other guidelines for the round robin in general are included in Appendix B.

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PART I

COMPARISON OF TWO ANALYTICAL TEST METHODS FOR THE DETERMINATION OF THE AMOUNT OF RDX IN COMPOSITION B.

This section evaluates the accuracy of each method by comparing the results obtained using a given analytical method with the known value of a synthetic sample. It sets 95 percent confidence limits on the means generated by each method using a student's "t" distribution. If the test is of the desired accuracy, the actual sample value should fall within the 95 percent confidence interval generated by each method. The average bias for each method with respect to the synthetic samples is also calculated.

Preparation of Synthetic Samples

The technician should weigh accurately to within $\pm .001g$ fourteen different synthetic samples of Composition B adhering to the specified composition (not including the tolerances). A 10.000g sample is used in the following examples. Thus the technician will have fourteen 10.000g samples having the following composition:

Amount of RDX	=	$5.950 \pm .001g$	
Amount of TNT	=	$3.950 \pm .001g$	synthetic sample 1
Amount of WAX	=	$.100 \pm .001g$	

In addition to these samples, eight other samples should be made with four having the amount of RDX at a low concentration of RDX:

$5.000 \pm .001g$ RDX	
$4.900 \pm .001g$ TNT	synthetic sample 2
$.100 \pm .001g$ WAX	

and four samples are prepared at a high concentration of RDX:

$7.000 \pm .001g$ RDX	
$2.900 \pm .001g$ TNT	synthetic sample 3
$.100 \pm .001g$ WAX	

It should be emphasized that all synthetic samples should be weighed out individually. Thus in preparing synthetic sample 1, fourteen weighings of each of the three components (42 separate weighings) are required.

In analyzing synthetic sample number 1, seven determinations using each method are required. In analyzing synthetic samples 2 and 3, duplicate determinations using each method are required. The results are shown in Tables 1 and 2.

TABLE 1

Data Reported for RDX Content
in 14 Synthetic Samples by 2 Methods

Synthetic Sample 1:

<u>Method 1 (specified method)</u>	<u>Method 2 (proposed method)</u>
5.960	5.979
5.975	6.068
5.991	5.916
5.966	6.224
5.936	6.089
5.988	6.193
5.892	5.994
$\bar{X} = 5.960$	$\bar{X} = 6.066$
$S = .0363$	$S = .1131$

NOTE: All data has been inspected for outliers and none are present.

TABLE 2

Data Reported for RDX Content
in 8 Synthetic Samples By 2 Methods

Synthetic Sample 2:

<u>Method 1</u>	<u>Method 2</u>
5.037	5.158
5.006	5.058
$\bar{X} = 5.022$	$\bar{X} = 5.108$
$S = .0218$	$S = .0707$

Synthetic Sample 3:

<u>Method 1</u>	<u>Method 2</u>
6.991	7.058
7.043	7.152
$\bar{X} = 7.017$	$\bar{X} = 7.105$
$S = .0368$	$S = .0665$

95% Confidence Limits for the Mean

A two sided "t" test is used to set confidence limits on the means generated by each method. From Table A-2 for 6 degrees of freedom, $t_{.975} = 2.447$. The 95% confidence limits on μ are given by:

$$(\bar{X} - ts/n^{.5}) \leq \mu \leq (\bar{X} + ts/n^{.5}) \quad \text{where } t = t_{.975}$$

Method 1

$$ts/n^{.5} = \frac{2.447 (.0363)}{(7)^{.5}} = .0336$$

$$\bar{X} = 5.960$$

$$5.926 \leq \mu \leq 5.994$$

Method 2

$$ts/n^{.5} = \frac{2.447 (.1131)}{(7)^{.5}}$$

$$\bar{X} = 6.066$$

$$5.961 \leq \mu \leq 6.170$$

Conclusions

The actual value of the sample composition ($\mu = 5.950$) is included in the 95% confidence interval generated by Method 1 and is not included in the 95% confidence interval generated by Method 2.

Determination of the Average Bias

The bias is the difference between the measured and actual value:

$$\begin{aligned}\text{Bias} &= \text{Measured Value} - \text{Actual value} \\ &= \bar{X} - \mu\end{aligned}$$

For each method a bias is calculated for each synthetic sample. The weighted average bias is then calculated.

Method 1

Synthetic Sample 1	Bias = $\bar{X} - \mu$ $\approx 5.960 - 5.950$ $\approx .010$	n = 7 replicates
Synthetic Sample 2	Bias $\approx 5.022 - 5.00$ $\approx .022$	n = 2 replicates
Synthetic Sample 3	Bias $\approx 7.017 - 7.00$ $\approx .017$	n = 2 replicates
Weighted average bias =	$\frac{7 (.010) + 2 (.022) + 2 (.017)}{11} = .0135$	

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Method 2

Synthetic Sample 1	Bias = $6.066 - 5.950$ $= .116$	n = 7 replicates
Synthetic Sample 2	Bias = $5.108 - 5.000$ $= .108$	n = 2 replicates
Synthetic Sample 3	Bias = $7.105 - 7.000$ $= .105$	n = 2 replicates
Weighted average bias =	$\frac{7 (.116) + 2 (.108) + 2 (.105)}{11} = .113$	

11

Conclusions

The weighted average bias is about 10 times greater for Method 2 than for Method 1.

PART 2

TREATMENT OF DATA

This section uses Cochran's Test to determine the homogeneity of the variances obtained among lots for a given method. In order to determine the presence of outliers Dixon's Range Test is used. Finally the means and variances of each method are compared using "F" tests and "t" tests.

Dixon's Range Test

The observed values are first arranged in increasing or decreasing order (see Table 3) and labeled X_1 to X_n (see Table 4). The ratio r_{11} is then calculated and compared with the critical values in Table A-1 at the .05 significance level. If the calculated value of r_{11} exceeds the value obtained from Table A-1, the point is considered an extreme observation and eliminated from the set of data.

TABLE 3

Data Reported for RDX Content in Three Different Lots
Using the Proposed Method and the Specified Method.

Method 2 (Proposed Method)			Method 1 (Specified Method)		
LOT A	LOT B	LOT C	LOT A	LOT B	LOT C
5.742	5.878	6.030	5.646	5.449	5.947
5.822	5.983	6.070	5.695	5.863	5.999
5.860	6.026	6.158	5.704	5.899	6.042
5.860	6.032	6.165	5.742	5.921	6.063
5.865	6.052	6.193	5.743	5.938	6.070
5.884	6.054	6.199	5.748	5.956	6.078
5.899	6.128	6.238	5.753	5.959	6.087
5.900	6.153	6.271	5.780	5.993	6.101
5.932	6.190	6.298	5.796	6.008	6.121
5.943	6.195	6.304	5.811	6.060	6.125
$\bar{X} =$ 5.861	$\bar{X} =$ 6.112	$\bar{X} =$ 5.193	$\bar{X} =$ 5.715	$\bar{X} =$ 5.905	$\bar{X} =$ 6.0633
S = .0578	S = .138	S = .0915	S = .0496	S = .169	S = .0553

TABLE 4

Data for Lot B Method 1 Arranged in Increasing Order
with Points Labeled for Dixons's Range Test

	(increasing order)	(decreasing order)
5.449	X ₁	X ₁₀
5.863	X ₂	X ₉
5.899	X ₃	X ₈
5.921	X ₄	X ₇
5.938	X ₅	X ₆
5.956	X ₆	X ₅
5.959	X ₇	X ₄
5.993	X ₈	X ₃
6.008	X ₉	X ₂
6.060	X ₁₀	X ₁

DIXON'S RANGE TEST APPLIED TO LOT B METHOD 1

For increasing order:

$$r_{11} = (X_2 - X_1)/(X_9 - X_1) = (5.863 - 5.449)/(6.008 - 5.44) = .741$$

For decreasing order:

$$r_{11} = (X_2 - X_1)/(X_9 - X_1) = (6.008 - 6.060)/(5.863 - 6.060) = .264$$

From Table A-1 for 10 observations at .05 significance level, r_{11} should be rejected if it is greater than .477. Therefore, point X_1 in the increasing order list should be eliminated. (The point 5.449 is an outlier.) The data would then consist of the remaining 9 observations.

Data (Lot B Method 1) Extreme Observation Removed

5.863

5.899

5.921

5.938

5.956

5.959

5.993

6.008

6.060

$\bar{X} = 5.955$

$S = .0595$

All other data in Table 3 has been inspected for outliers and none are present.

Cochran's Test for Homogeneity of Variances

Cochran's test uses the equation:

$$C = \frac{\text{largest } s_i^2}{\sum s_i^2}$$

where s_i^2 is the variance of Lot A, B, or C for a given method

If C is less than the value exceeded 5 percent of the time (i.e., $C_{.95}$) there is no evidence to indicate the variances are not homogeneous.

For Method 2:

$$C = \frac{.138^2}{.058^2 + .138^2 + .0915^2} = .619$$

For Method 1:

$$C = \frac{.169^2}{.0496^2 + .169^2 + .0553^2} = .838$$

For 3 means ($k = 3$) and 9 degrees of freedom ($df = 9$) the value of C at the .05 significance level may be found in Table A-4.

$$C_{.95} = .6167 \quad (k = 3, df = 9)$$

Since both values of C calculated for Method 1 and Method 2 are larger than $C_{.95}$ we reject the hypothesis that the variances of lots A, B and C are homogeneous. Upon further inspection of the data it appears that the variance for Lot B is noticeably larger than the variance of lots A and C for both methods.

If we drop the data for Lot B and consider only lots A and C we can recalculate C for each method.

For Method 2:

$$C = \frac{.0915^2}{.058^2 + .0915^2} = .713$$

For Method 1:

$$C = \frac{.0553^2}{.0496^2 + .0553^2} = .554$$

For 2 means ($k = 2$) and 9 degrees of freedom ($df = 9$) the value of C at the .05 significance level may be found in Table A-4.

$$C_{.95} = .8010 \quad (k = 2, df = 9)$$

Therefore, since both calculated C's are less than .8010 we have no evidence to reject the hypothesis that the variances are homogeneous for lots A and C.

Determination of Equality of Means and Variances for
Methods 1 and Method 2

This section compares and contrasts the means and variances obtained by each method for a given lot. For each lot the following operations are performed:

a. An "F" test to determine if the variances of Method 1 and Method 2 are significantly different.

b. A "t" test to determine if the means generated by Method 1 and Method 2 are significantly different.

Lot A

	<u>Mean</u>	<u>Standard Deviation</u>
Method 1	5.715	.0496
Method 2	4.871	.0578

The F ratio is formed by computing the ratio of the sample variances:

$$(2-1) \quad F = (.0496)^2 / (.0578)^2 = .736$$

Critical values of F are determined from Appendix A.

The degrees of freedom for each mean is 9.

$$\text{therefore: } F_{.975} (9, 9) = 4.03$$

$$\text{and: } F_{.025} (9, 9) = .248$$

Since the calculated value of F (i.e., .736) is in between the two critical F values, we have no reason to suspect that the variances are different at the 5 percent level of significance.

As a result of this calculation, the variances for Method 1 and Method 2 may be pooled.

$$(2-2) \quad s_p^2 = ((n_1 - 1) s_1^2 + (n_2 - 1) s_2^2) / (n_1 + n_2 - 2)$$

Since $n_1 = n_2 = 10$, this reduces to:

$$s_p^2 = (s_1^2 + s_2^2) / 2$$

$$s_p^2 = (.04962 + .05782) / 2$$

$$s_p^2 = .005801/2$$

$$s_p^2 = .002901$$

$$s_p = .0539$$

Using the pooled standard deviation ($s_p = .0539$) obtained by pooling the standard deviations of Methods 1 and 2 for Lot A, a "t" score may be calculated to determine if the means generated by Method 1 and 2 are significantly different.

$$(2-3) \quad t = (\bar{X}_1 - \bar{X}_2) / s_p ((1/n_1) + (1/n_2))^{.5}$$

$$t = (5.715 - 5.871) / .0539 \times (1/10 + 1/10)^{.5}$$

$$t = - 6.47$$

This statistic should have a "t" distribution with $(n_1 + n_2 - 2)$ or eighteen degrees of freedom.

$$t_{.975} (18) = 2.101$$

$$t_{.025} (18) = - 2.101$$

Since "t" calculated is less than -2.101 we can say the means generated by method 1 and 2 are different at the 5 percent level of significance.

Similar calculations should be performed on lots B and C. For the case when the variances for Methods 1 and 2 are found to be nonhomogeneous the "t" score is calculated from:

$$(2-4) \quad t = (x_1 - x_2) / (s_1^2/N_1 + s_2^2/N_2)^{.5}$$

with the degrees of freedom calculated from

$$(2-5) \quad df = \frac{\frac{(s_1^2/N_1 + s_2^2/N_2)^2}{(s_1^2/N_1)^2 + (s_2^2/N_2)^2} - 2}{\frac{1}{N_1 + 1} + \frac{1}{N_2 + 1}}$$

The results of these calculations for Lots B and C are stated below:

Lot B. Variances and means are significantly different for each method.

Lot C. Variances are homogeneous for methods 1 and 2 but means are significantly different.

Summary of Results Obtained in Part 2

1. Cochran's test for homogeneity of variances has indicated that the variances are homogeneous for each method only if the data for Lot B is not included. The use of Dixon's range test for identifying an "outlier" has been illustrated using the data for Lot B, Method 1. The results of the test indicated that an outlier did exist in the data. These two results seem to indicate that Lot B may be highly variable and perhaps heavier tailed than a normal distribution.

2. The means and variances obtained for each lot using Methods 1 and 2 have been contrasted and compared for the three lots in question. The variances appear to be homogeneous for two of the lots and nonhomogeneous for one lot. In all lots the means generated by the two methods have been shown to be significantly different using the "t" distribution.

3. The results obtained in Parts 1 and 2 have indicated that the proposed method is less accurate than the specified method. The variances of each method appear to be homogeneous except for the results obtained with Lot B. However, if the variances in Tables 2 and 3 are analyzed using an F test (as on Page 11) there is evidence to indicate the variances may not be homogeneous. In order to determine if the variances are homogeneous for both methods additional data is required. The fact that Method 2 appears to exhibit a constant bias may be useful, since in some cases the bias may be of such a nature that it is predictable and can be used in characterizing the method.

At this point a decision must be made in regard to running the round robin. The data which has been evaluated suggests that the proposed method will not be an equivalent method. Based on these results, it is probably not worthwhile to conduct the round robin, however, in order to continue the example it is decided to run the round robin. The results are shown in Parts 3 and 4.

PART 3

EVALUATION OF THE REPEATABILITY AND REPRODUCIBILITY OF EACH METHOD

In this section the bulk of the data is treated in order to get estimates of the method repeatabilities and reproducibilities. The data for three lots is analyzed by five laboratories for each method. This data along with the averages and standard deviations of each lab is presented in Appendix C.

In order to treat this data the following assumptions or tests must be made:

- a. The data is normally distributed.
- b. The data for each laboratory has been inspected for outliers and none are present.
- c. The data for a given method and a given lot (data from five labs) have been examined for homogeneity of variance, and the results have shown that the variances are indeed homogeneous.

The basic approach used in this section is to use the technique of analysis of variance (ANOVA) to determine the variance between the means and the variance within each lab. An "F" test is used to compare the two variances. If the "F" value is found to be significant, the repeatability and reproducibility are calculated by two different equations. If there is no significant difference found by application of an F test, the repeatability is equal to the reproducibility.

The calculations involved in this procedure are illustrated in a stepwise fashion for the benefit of the uninformed reader. The results of analysis of variance performed on the six sets of data are shown in Appendix D. The method repeatabilities and/or reproducibilities are presented in Table 6.

Definitions Having to do with Repeatability and Reproducibility

Laboratory - A laboratory must consist of a single analyst.

Test result - An average of n replicate measurements

Repeatability - A quantity that will be exceeded only about 5 percent of the time by the difference, taken in absolute value, of two randomly selected test results obtained in the same laboratory on a given material.

reproducibility - A quantity that will be exceeded only about 5 percent of the time by the difference, taken in absolute value, of two single test results made on the same material in two different, randomly selected laboratories.

Calculations Required for Analysis of Variance Using a One Way Classification

The form of the data is presented in Table 5.

In order to perform a one way analysis of variance, the following terms must be evaluated:

$$\sum \sum X_{ij}^2, \sum (T_{i+}^2/n_i), T_{++}^2/N$$

The pooled variance is calculated as:

$$S_p^2 = (\sum \sum X_{ij}^2 - \sum (T_{i+}^2/n_i)) / (\sum n_i - k)$$

The mean square between categories is calculated as:

$$S_m^2 = \frac{\sum (T_{i+}^2/n_i) - T_{++}^2/N}{k - 1}$$

where

- k = Number of categories
- n_i = Number of observations in each category
- T_{i+} = Sum of the observations in a category
- N = Total number of observations
- S_p² = Within groups mean square (or pooled variances)
- S_m² = Between groups mean square

S_p = Pooled standard deviation
 df = Degrees of freedom for each sum of squares
 X_{ij} = Observation number i from column j
 $\sum X_{ij}^2$ = The sum of the squares of all the data points in a given group of data.
 T_{++} = Sum of all the observations
 \bar{X} = Grand mean = T_{++}/N
 \bar{X}_i = Mean of each category = T_{i+}/n
 SS = Sum of squares
 MS = Mean square

The results of these calculations are then presented in a table such as Table 6.

TABLE 5 FORM OF THE DATA FOR ONE WAY ANOVA

LABORATORY				
A	B	C	D	E
X ₁₁	X ₂₁	X ₃₁	X ₄₁	X ₅₁
X ₁₂	X ₂₂	X ₃₂	X ₄₂	X ₅₂
X ₁₃	X ₂₃	X ₃₃	X ₄₃	X ₅₃
.
.
.
X ₁₁₀	X ₂₁₀	X ₃₁₀	X ₄₁₀	X ₅₁₀
TOTAL:	T ₁₊	T ₂₊	T ₃₊	T ₄₊
MEAN:	\bar{X}_1	\bar{X}_2	\bar{X}_3	\bar{X}_4
				\bar{X}_5

$$T_{1+} = X_{11} + X_{12} + X_{13} + X_{14} + X_{15}$$

$$T_{++} = T_{1+} + T_{2+} + T_{3+} + T_{4+} + T_{5+}$$

$$\bar{X}_1 = T_{1+}/10, \text{ etc}$$

$$\bar{X} = T_{++}/50$$

TABLE 6 - One Way ANOVA

	Sum of Squares	df	Mean Square	Estimate of
Between	$\Sigma(T_{i+}^2/n_i) - T_{++}^2/N$	k-1	s_M^2	$\sigma^2 + n\sigma_m^2$
Within	$\Sigma \Sigma X_{ij}^2 - \Sigma T_{i+}^2/n_i$	N-k	s_p^2	σ^2
Total	$\Sigma \Sigma X_{ij}^2 - T_{++}^2/N$			

In order to illustrate the calculation of the quantities presented in Table 6, sample calculations are performed using the data for lot C method 2. The data is presented below and the calculations using this data are shown on the next page.

DATA FOR LOT C METHOD 2

	LAB A	LAB B	LAB C	LAB D	LAB E
	6.0330	6.1590	6.0040	5.9130	6.2040
	6.1450	6.0700	6.1420	6.0640	6.0480
	6.0630	6.0170	6.0530	5.9430	6.0060
	6.1250	6.2400	5.9430	5.9030	6.2140
	6.1940	6.0220	6.0540	6.0720	6.1970
	6.1380	6.2770	6.2620	6.0470	6.2330
	6.0640	6.1610	6.1350	6.1820	6.0740
	5.8790	5.9950	5.9910	6.0360	6.2200
	6.1630	6.1980	5.8450	6.0290	5.9970
	6.1660	6.3170	6.0550	5.9350	6.0360
Lab Totals:	61.015	61.534	60.500	60.134	61.414

Each individual observation by the five labs is squared and these values are summed:

$$\begin{aligned} (3-1) \sum x_{ij}^2 &= 6.089^2 + 6.145^2 + 6.063^2 + \dots + 5.997^2 + 6.086^2 \\ &= 1856.17116 \end{aligned}$$

The five lab totals are squared, added together and then divided by the number of replicates per lab.

$$\begin{aligned} (3-2) \sum T_{i.}^2 / n_i &= (61.05^2 + 61.534^2 + \dots + 61.414^2) / 10 \\ &= 1855.72907 \end{aligned}$$

The grand total is squared and divided by the total number of replicates.

$$\begin{aligned} (3-3) T_{++}^2 / N &= (61.015 + 61.534 + \dots + 61.414)^2 / 50 \\ &= 1855.586648 \end{aligned}$$

The degrees of freedom used for the between groups mean square is

$$\begin{aligned} (3-4) df_1 &= k - 1 \\ &= 5 - 1 \\ &= 4 \end{aligned}$$

The degrees of freedom used for the within groups mean square

$$\begin{aligned} (3-5) df_2 &= N - k \\ &= 50 - 5 \\ &= 45 \end{aligned}$$

Using the results of (3-1), (3-2), and (3-3) the Sum of Squares between groups and within groups may be evaluated.

$$(3-6) \text{ Sum of Squares between groups} = \sum T_{i+}^2 / n_i - T_{++}^2 / N$$

$$= 1855.72907 - 1855.586648$$

$$= .14243$$

$$(3-7) \text{ Sum of Squares within groups} = \sum \sum x_{ij}^2 - \sum T_{i+}^2 / n_i$$

$$= 1856.17116 - 1855.72907$$

$$= .44209$$

Using the results obtained in equations (3-4) through (3-7), the two mean squares are now evaluated:

$$(3-8) \text{ Mean Square between groups} = (\sum T_{i+}^2 / n_i - T_{++}^2 / N) / (k - 1)$$

$$= \frac{.14243}{4}$$

$$= 3.5606 \times 10^{-2}$$

$$(3-9) \text{ Mean Square within groups} = (\sum \sum x_{ij}^2 - \sum T_{i+}^2 / n_i) / (N - k)$$

$$= \frac{.44209}{45}$$

$$= 9.8242 \times 10^{-3}$$

The total Sum of Squares and degrees of freedom, df, may be calculated as the sum of the within groups and between groups contributions.

$$(3-10) \text{ Total Sum of Squares} = .14243 + .44209$$

$$(3-11) \text{ Total degrees of freedom} = 4 + 45 = 49$$

The F value is calculated by dividing the mean square between groups by the mean square within groups.

(3-12)

$$F = S_m^2 / S_p^2$$

$$F = (3.5606 \times 10^{-2}) / (9.8242 \times 10^{-3})$$

$$F = 3.624$$

The calculated "F" value is compared with the "F" distribution in Appendix A-3. "F" is presented as a function of df_1 and df_2 (the degrees of freedom of S_m^2 and S_p^2) and of the level of significance chosen. For this paper the .05 significance level is chosen. Therefore $F_{.95}(df_1, df_2)$ becomes $F_{.95}(4, 45)$ where $df_1 = 4$ (the degrees of freedom associated with S_m^2) and $df_2 = 45$ (the degrees of freedom associated with S_p^2).

$$F_{.95}(4, 45) = 2.59$$

Since the calculated "F" value (3.624) is greater than $F_{.95}(4, 45)$ we may conclude that the means of the 5 labs are unequal at the .05 significance level and that the reproducibility is different than the repeatability.

A computer program has been developed to do all calculations described in this section. Sample programs are shown in Appendix E.

The results of these calculations are then presented in Table 7 which has the form of Table 6. The results of each analysis of variance are presented in Appendix D. The grand mean of each data set is presented with each analysis of variance.

TABLE -
ANALYSIS OF VARIANCE FOR LOT C METHOD 2

	SS	DF	MS	F
BETWEEN	.142425	4	.035606	3.624338
WITHIN	.442090	45	.009824	
TOTAL	.584515	49	.011929	

THE GRAND MEAN FOR LOT C METHOD 2 IS 6.092

Evaluation of Repeatability and Reproducibility
Using Components of Variance

In order to evaluate the method reproducibility and repeatability it is necessary to break down S_m^2 and S_p^2 into their components of variance. This is done by utilizing the following equations.

$$(3-13) \quad S_p^2 = \sigma^2$$

$$(3-14) \quad S_m^2 = \sigma^2 + n\sigma_m^2$$

where σ^2 is an estimate of the population variance and σ_m^2 is an estimate of the variance of the mean of each lab about the grand mean.

Solving for σ^2 and σ_m^2 for this example:

$$\sigma^2 = 9.8242 \times 10^{-3}$$

$$\sigma^2 + n\sigma_m^2 = 3.5606 \times 10^{-2}$$

$$\sigma_m^2 = \frac{3.5606 \times 10^{-2} - 9.8242 \times 10^{-3}}{10}$$

$$\sigma_m^2 = 2.5782 \times 10^{-3}$$

The repeatability and reproducibility may now be calculated by use of equations (3-15) and (3-16):

$$(3-15) \quad \text{Repeatability} = 2.77 \times (\sigma^2/n)^{.5}$$

$$(3-16) \quad \text{Reproducibility} = 2.77 \times (\sigma^2 + \sigma_m^2/n)^{.5}$$

Solving these equations we find:

$$\begin{aligned} \text{Repeatability} &= 2.77 \times (9.8242 \times 10^{-3}/10)^{.5} \\ &= .0868 \end{aligned}$$

$$\begin{aligned} \text{and Reproducibility} &= 2.77 \times (2.5782 \times 10^{-3} + 9.8242 \times 10^{-3}/10)^{.5} \\ &= .1653 \end{aligned}$$

The remaining repeatabilities and reproducibilities are calculated in Appendix E. The results of these calculations are presented in Table 8.

TABLE 8

Repeatabilities and Reproducibilities as Calculated
Using a One Way Classification

	<u>Method 1</u>	
	<u>Repeatability</u>	<u>Reproducibility</u>
Lot A	.0397	.0397
Lot B	.0236	.0236
Lot C	.0454	.0454

	<u>Method 2</u>	
	<u>Repeatability</u>	<u>Reproducibility</u>
Lot A	.0869	.1645
Lot B	.0804	.1547
Lot C	.0868	.1653

PART 4

A SINGLE ESTIMATE OF THE REPRODUCIBILITY AND REPEATABILITY FOR EACH METHOD USING A TWO WAY CLASSIFICATION, ANALYSIS OF VARIANCE TECHNIQUE

In this section a method of combining the data for all three lots for a given method is illustrated. This can only be done if, for a given method, the variances for all the lots are homogeneous. It is assumed in this section that all variances are homogeneous.

Once the variances for all the lots (15 test results in all; that is three lots with each lot analyzed by five different laboratories) have been shown to be homogeneous, a single estimate of repeatability and reproducibility based on all the data points for that particular method is possible.

This is done with a two way classification analysis of variance technique.

The Analysis of Variance Table for a Two Way classification is shown in Table 9. The nomenclature used in Table 9 is described below:

n	= Number of replicates
r	= Number of rows
c	= Number of columns
\bar{X}_i	= Mean of each column
\bar{X}_j	= Mean of each row
\bar{X}_{ij}	= Individual mean of column i row j
\bar{X}	= Grand mean of all data points
\bar{X}_{ije}	= Each individual data point
S_c	= Sum of Squares for Columns
S_r	= Sum of Squares for Rows
S_I	= Sum of Squares for Interaction
S_w	= Sum of Squares for Within
S_T	= Sum of Squares for Total
S_S	= Sum of Squares for Subtotal

TABLE 1: TWO WAY ANALYSIS OF VARIANCE

	SUM OF SQUARES	df	MEAN SQUARE	ESTIMATE OF
Column Means	$rn\sum(\bar{X}_i - \bar{X})^2 = S_C$	c-1	$S_C/(c-1)$	$\sigma^2 + n\sigma_I^2 + cn\sigma_C^2$
Row Means	$cn\sum(\bar{X}_j - \bar{X})^2 = S_r$	r-1	$S_r/(r-1)$	$\sigma^2 + n\sigma_I^2 + rn\sigma_r^2$
Interaction	$S_g - S_C - S_r = S_I$	$(c-1)*(r-1)$	$S_I/(c-1)*(r-1)$	$\sigma^2 + n\sigma_I^2$
Subtotal	$n\sum\sum(\bar{X}_{ij} - \bar{X})^2 = S_g$	rc-1		
Within	$S_T - S_g = S_w$	rc(n-1)	$S_w/(rc(n-1))$	σ^2
Total	$\sum\sum\sum(X_{ije} - \bar{X})^2 = S_T$	rcn - 1		

In order to perform the calculations involved in computing a two way analysis of variance, it is necessary to arrange the data in two classifications. The data for method one is shown in Table 10. Here, the vertical classifications are the labs, and the horizontal classifications are the lots. Each set of data for one lab and one lot is referred to as a cell. Each cell contains ten replicates.

In addition to presenting the data in this fashion, it is also convenient to form a second table which shows the totals and subtotals of each row and column. The subtotals are actually the cell totals (i.e., the sum of ten data points for a given lot and a given lab). From these subtotals, the totals of each row and column are then formed. The totals and subtotals formed from the data in Table 10 are shown in Table 11.

Using a two way analysis of variance enables the statistician to separate the total variance into the components due to differences between labs and differences between lots. Since we do not want to include the variance due to differences between lots in our overall estimates of repeatability and reproducibility, this technique is useful to us. It enables us to combine the data from all three lots and thus get estimates of the relevant variances with higher degrees of freedom than with the single classification ANOVA. It also gives us a single estimate of repeatability and reproducibility for each test method.

The following example illustrates the calculations which must be performed on these arrays in order to evaluate the two-way analysis of variance. Once this has been determined, use is made of the components of variance approach to evaluate the appropriate components of variance in order to determine the method repeatability and reproducibility.

Calculations Required for Analysis of Variance Using a Two Way Classification

Using the data in Tables 10 and 11 the following calculations are performed:

(4-1) Sum of Squares for Total:

$$S_T = 5.776^2 + 5.730^2 + \dots + 6.029^2 + 5.990^2 - 883.757^2/150$$

$$S_T = 5208.5487 - 5206.8429$$

$$S_T = 1.7058$$

TABLE 1

TWO WAY CLASSIFICATION-DATA OF METHOD 1

	LAB A	LAB B	LAB C	LAB D	LAB E
LOT A	7763	7582	8270	7690	7790
	557703	557703	557703	557703	558400
	7773	7582	557703	7773	557703
	7773	558400	557703	7773	557703
	7773	558400	557703	7773	557703
	7773	558400	557703	7773	557703
	7773	558400	557703	7773	557703
	7773	558400	557703	7773	557703
	7773	558400	557703	7773	557703
	7773	558400	557703	7773	557703
LOT B	7763	7582	8270	7690	7790
	557703	557703	557703	557703	558400
	7773	7582	557703	7773	557703
	7773	558400	557703	7773	557703
	7773	558400	557703	7773	557703
	7773	558400	557703	7773	557703
	7773	558400	557703	7773	557703
	7773	558400	557703	7773	557703
	7773	558400	557703	7773	557703
	7773	558400	557703	7773	557703
LOT C	7763	7582	8270	7690	7790
	557703	557703	557703	557703	558400
	7773	7582	557703	7773	557703
	7773	558400	557703	7773	557703
	7773	558400	557703	7773	557703
	7773	558400	557703	7773	557703
	7773	558400	557703	7773	557703
	7773	558400	557703	7773	557703
	7773	558400	557703	7773	557703
	7773	558400	557703	7773	557703

TABLE 11: DATA OF METHOD 1-TOTALS AND SUBTOTALS

LAB A	LAB B	LAB C	LAB D	LAB E	
57.4520	57.7050	57.5040	57.6160	57.7340	288.0110
59.2510	59.4160	59.2000	59.2930	59.0880	296.2480
59.9960	59.7530	60.0980	59.7510	59.9000	299.4980
176.6990	176.8740	176.8020	176.6600	176.7220	883.7570

(4-2) Sum of Squares for Subtotal:

$$S_S = 57.4520^2/10 + 59.2510^2/10 + \dots + 59.0880^2/10 + 59.9000^2/10 - 883.757^2/150$$

$$S_S = 5208.26638 - 5206.8429$$

$$S_S = 1.42348$$

(4-3) Sum of Squares for Rows:

$$S_R = 288.0110^2/50 + 296.2480^2/50 + 299.4980^2/50 - 883.757^2/150$$

$$S_R = 5208.24531 - 5206.8429$$

$$S_R = 1.4024$$

(4-4) Sum of Squares for Columns:

$$S_C = 176.6990^2/30 + 176.8740^2/30 + \dots + 176.7220^2/30 - 883.757^2/150$$

$$S_C = 5206.84388 - 5206.8429$$

$$S_C = 9.8666 \times 10^{-4}$$

(4-5) Sum of Squares for Within:

This value is found by subtracting the Sum of Squares for the subtotal from the Sum of Squares for the total.

$$SS \text{ for within} = 1.7058 - 1.42348 = .2823$$

(4-6) Sum of Squares for Interactions

This value is found by subtracting the sum of Squares for Columns plus the Sum of Squares for rows from the Sum of Squares for the subtotal.

$$SS \text{ for Interactions} = 1.42348 - 1.4024 - 9.866 \times 10^{-4} = .0201$$

Table 12:

TWO WAY ANOVA-METHOD 1

	SS	DF	MS	F-RATIO
ROWS	1.4024	2	.70121	279.2823
COLUMNS	.0010	4	.00025	.0981
INTERACTIONS	.0201	8	.00251	1.2007
WITHIN	.2823	135		
TOTALS	1.7058	149	.00209	

Table 13:

TWO WAY ANOVA-METHOD 2

	SS	DF	MS	F-RATIO
ROWS	1.2357	2	.61786	17.4876
COLUMNS	.1256	4	.03141	.8889
INTERACTIONS	.2827	8	.03533	3.7729
WITHIN	1.2642	135	.00936	
TOTALS	2.9082	149		

The repeatability is then calculated as

$$(4-14) \text{ Repeatability} = 2.77 \times (\sigma^2/n)^{.5}$$

The reproducibility is calculated as

$$(4-15) \text{ Reproducibility} = 2.77 \times (\sigma^2/n + \sigma_C^2 + \sigma_I^2)^{.5}$$

Note the lot to lot variance, σ_I^2 , is not included in the variance estimate used for reproducibility.

Components of Variance Estimates Using Two-way ANOVA for Method I

Equations (4-13), (4-12) and (4-10) may be solved simultaneously to furnish estimates of the components of variance used in determining the repeatability and reproducibility from equations (4-14) and (4-15). The procedure follows.

The repeatability variance is estimated using the mean square for within from equation (4-13):

$$\sigma^2_{\text{repeatability}} = \sigma^2 = .00209 \quad (4-16)$$

The interaction variance is estimated using the mean square for interactions defined in equation (4-12) and using the value of σ^2 which was determined in equation (4-16):

$$\sigma^2_{I+I^2} = .00251 \quad (4-17)$$

where $n = 10$, $\sigma^2 = .00209$

$$\sigma_I^2 = (.00251 - .00209)/10$$

$$\sigma_I^2 = 4.20 \times 10^{-5} \quad (14-18)$$

The variance due to differences between labs is estimated using the mean square for columns defined in equation (4-10) and using the values of σ^2 and σ_I^2 as defined by equation (4-16) and (4-18):

$$\begin{aligned}\sigma^2 + n\sigma_I^2 + rn\sigma_C^2 &= .00025 \\ \text{where } r &= 3, n = 10, \sigma^2 + n\sigma_I^2 = .00251 \\ \sigma_C^2 &= (.0025 - .00251)/30 \\ \sigma_C^2 &= -7.533 \times 10^{-5} \quad (4-19)\end{aligned}$$

The sum of the interaction component of variance and the lab to lab variance is then calculated

$$\begin{aligned}\sigma_C^2 + \sigma_I^2 &= 4.2 \times 10^{-5} + (-7.533 \times 10^{-5}) \\ \sigma_C^2 + \sigma_I^2 &= -3.33 \times 10^{-5} \quad (4-20)\end{aligned}$$

Since $\sigma_C^2 + \sigma_I^2 < 0$, the reproducibility is equal to the repeatability. The rationale for this is the following:

If either the between-laboratory or interaction variances is not significant, the existence of random error may cause either of the respective mean square ratios to be less than one. If this occurs the values σ_C^2 or σ_I^2 may result in negative quantities. Method reproducibility may still be calculated, unless the sum of $\sigma_C^2 + \sigma_I^2$ is negative, in which case the whole of the variation may be assumed to be due to replication error (σ^2) and the reproducibility is equal to the repeatability.

In view of this fact for Method 1,

$$\sigma^2 \text{ reproducibility} = \sigma^2 \text{ repeatability} = \sigma^2 = .00209$$

and the repeatability is calculated from equation (4-14).

$$\begin{aligned} \text{Repeatability} &= 2.77 \times (\sigma^2/n)^{.5} \\ &= 2.77 \times (.00209/10)^{.5} \\ &= .0400 \end{aligned}$$

$$\text{Reproducibility} = \text{Repeatability} = .0400$$

The same procedure is now repeated for Method 2.

Components of Variance Calculations for Method 2 Using Data from Table 13

Estimate of σ^2 :

$$\sigma^2 = .00936$$

Estimate of σ_I^2 :

$$\sigma^2 + n\sigma_I^2 = .0353$$

$$\sigma_I^2 = \frac{.03533 - .00936}{10}$$

$$\sigma_I^2 = .002597$$

Estimate of σ_C^2 :

$$\sigma^2 + n\sigma_I^2 + rn\sigma_C^2 = .03141$$

$$\sigma_C^2 = \frac{.03141 - .03533}{30}$$

$$\sigma_C^2 = -1.306 \times 10^{-4}$$

Now the repeatability and reproducibility may be calculated from equations (4-14) and (4-15).

$$\text{Repeatability} = 2.77 \times (\sigma^2/n)^{.5}$$

$$= 2.77 \times (.00936/n)^{.5} \quad n = 10$$

$$= 2.77 \times (.00936/10)^{.5}$$

$$= .0847$$

$$\text{Reproducibility} = 2.77 \times (\sigma^2/n + \sigma_c^2 + \sigma_f^2)^{.5}$$

$$= 2.77 \times (.00936/10 + (-1.306 \times 10^{-4}) + (.002597))^{.5}$$

$$= .162$$

The results of these calculations are summarized in Table 14.

TABLE 14

Repeatabilities and Reproducibilities
as Calculated Using a Two way Classification

Method 1	
Repeatability	Reproducibility
.0400	.0400
Method 2	
Repeatability	Reproducibility
.0847	.162

PART 5

CONCLUSIONS

1. Method 1 is more accurate than Method 2. This has been demonstrated in Part 1.
2. The repeatability of Method 2 is greater than the Repeatability of Method 1 (Parts 3 and 4).
3. The lab to lab variation is significant for labs using Method 2 but not significant for labs using Method 1 (Parts 3 and 4).
4. In view of 2 and 3 no attempt has been made to characterize the bias exhibited by Method 2.
5. Method 2 is not equivalent to Method 1.

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APPENDIX A
Statistical Tables Used in this Document

TABLE A-1

TABLE A-8. SIMPLIFIED STATISTICS (Continued)
8c. Criteria for Testing for Extreme Mean

Statistic	Number of obs., k	Critical values						
		$\alpha = .30$	$\alpha = .20$	$\alpha = .10$	$\alpha = .05$	$\alpha = .02$	$\alpha = .01$	$\alpha = .005$
$r_{10} = \frac{X_2 - X_1}{X_k - X_1}$	3	.684	.781	.886	.941	.976	.983	.994
	4	.471	.560	.679	.765	.816	.889	.926
	5	.373	.451	.557	.642	.729	.780	.821
	6	.318	.386	.482	.560	.644	.698	.740
	7	.281	.341	.434	.507	.586	.637	.680
$r_{11} = \frac{X_2 - X_1}{X_{k-1} - X_1}$	8	.318	.385	.479	.554	.631	.683	.725
	9	.288	.352	.441	.512	.587	.635	.677
	10	.265	.325	.409	.477	.551	.597	.639
$r_{12} = \frac{X_2 - X_1}{X_{k-2} - X_1}$	11	.391	.442	.517	.576	.638	.679	.713
	12	.370	.419	.490	.546	.605	.642	.675
	13	.351	.399	.467	.521	.578	.615	.649
$r_{13} = \frac{X_2 - X_1}{X_{k-3} - X_1}$	14	.370	.421	.492	.546	.602	.641	.674
	15	.353	.402	.472	.525	.579	.616	.647
	16	.338	.386	.454	.507	.559	.595	.624
	17	.325	.373	.438	.490	.542	.577	.605
	18	.314	.361	.424	.475	.527	.561	.589
	19	.304	.350	.412	.462	.514	.547	.575
	20	.295	.340	.401	.450	.502	.535	.562
	21	.287	.331	.391	.440	.491	.524	.551
	22	.280	.323	.382	.430	.481	.514	.541
	23	.274	.316	.374	.421	.472	.505	.532
	24	.268	.310	.367	.413	.464	.497	.524
	25	.262	.304	.360	.406	.457	.489	.516

TABLE A-2

TABLE A-5. PERCENTILES OF THE *t* DISTRIBUTION*

df	$t_{.90}$	$t_{.80}$	$t_{.70}$	$t_{.60}$	$t_{.50}$	$t_{.40}$	$t_{.30}$	$t_{.20}$	$t_{.10}$
1	.325	.727	1.376	3.078	6.314	12.706	31.821	63.657	
2	.289	.617	1.061	1.886	2.920	4.303	6.965	9.925	
3	.277	.584	.978	1.638	2.353	3.182	4.541	5.811	
4	.271	.569	.941	1.533	2.132	2.776	3.747	4.604	
5	.267	.559	.920	1.476	2.015	2.571	3.365	4.032	
6	.265	.553	.906	1.440	1.913	2.417	3.143	3.707	
7	.263	.549	.896	1.415	1.895	2.365	2.998	3.499	
8	.262	.546	.889	1.397	1.860	2.306	2.896	3.355	
9	.261	.543	.883	1.383	1.833	2.262	2.821	3.250	
10	.260	.542	.879	1.372	1.812	2.228	2.764	3.169	
11	.260	.540	.876	1.363	1.796	2.201	2.718	3.106	
12	.259	.539	.873	1.356	1.782	2.179	2.681	3.055	
13	.259	.538	.870	1.350	1.771	2.160	2.650	3.012	
14	.258	.537	.868	1.345	1.761	2.145	2.624	2.977	
15	.258	.536	.866	1.341	1.753	2.131	2.602	2.947	
16	.258	.535	.865	1.337	1.746	2.120	2.583	2.921	
17	.257	.534	.863	1.333	1.740	2.110	2.567	2.898	
18	.257	.534	.862	1.330	1.734	2.101	2.552	2.878	
19	.257	.533	.861	1.328	1.729	2.093	2.539	2.861	
20	.257	.533	.860	1.325	1.725	2.086	2.528	2.845	
21	.257	.532	.859	1.323	1.721	2.080	2.518	2.831	
22	.256	.532	.858	1.321	1.717	2.074	2.508	2.819	
23	.256	.532	.858	1.319	1.714	2.069	2.500	2.807	
24	.256	.531	.857	1.318	1.711	2.064	2.492	2.797	
25	.256	.531	.856	1.316	1.708	2.060	2.485	2.787	
26	.256	.531	.856	1.315	1.706	2.056	2.479	2.779	
27	.256	.531	.855	1.314	1.703	2.052	2.473	2.771	
28	.256	.530	.855	1.313	1.701	2.048	2.467	2.763	
29	.256	.530	.854	1.311	1.699	2.045	2.462	2.756	
30	.256	.530	.854	1.310	1.697	2.042	2.457	2.750	
40	.255	.529	.851	1.303	1.684	2.021	2.423	2.704	
60	.254	.527	.848	1.296	1.671	2.000	2.390	2.660	
120	.254	.526	.845	1.289	1.658	1.980	2.358	2.617	
∞	.253	.524	.842	1.282	1.645	1.960	2.326	2.576	
df	$-t_{.90}$	$-t_{.80}$	$-t_{.70}$	$-t_{.60}$	$-t_{.50}$	$-t_{.40}$	$-t_{.30}$	$-t_{.20}$	$-t_{.10}$

TABLE A-3

TABLE A-7a. F DISTRIBUTION, UPPER 5% POINTS ($F_{.05}$)

		Degrees of freedom for numerator																		
		1	2	3	4	5	6	7	8	9	10	12	15	20	24	30	40	60	120	∞
Degrees of freedom for denominator	1	161	200	216	225	230	234	237	239	241	242	244	246	248	249	250	251	252	253	254
	2	18.5	19.0	19.2	19.2	19.3	19.3	19.4	19.4	19.4	19.4	19.4	19.4	19.4	19.5	19.5	19.5	19.5	19.5	19.5
	3	10.1	9.55	9.28	9.12	9.01	8.94	8.89	8.85	8.81	8.79	8.74	8.70	8.66	8.64	8.62	8.59	8.57	8.55	8.53
	4	7.71	6.94	6.59	6.39	6.26	6.16	6.09	6.01	6.00	5.96	5.91	5.86	5.80	5.77	5.75	5.72	5.69	5.66	5.63
	5	6.61	5.79	5.41	5.19	5.05	4.95	4.88	4.82	4.77	4.74	4.68	4.62	4.56	4.53	4.50	4.46	4.43	4.40	4.37
	6	5.99	5.14	4.76	4.53	4.39	4.28	4.21	4.15	4.10	4.06	4.00	3.94	3.87	3.81	3.77	3.74	3.70	3.67	3.64
	7	5.59	4.74	4.35	4.12	3.97	3.87	3.79	3.73	3.68	3.64	3.57	3.51	3.44	3.41	3.38	3.34	3.30	3.27	3.23
	8	5.32	4.46	4.07	3.84	3.69	3.58	3.50	3.44	3.39	3.35	3.28	3.22	3.15	3.12	3.08	3.04	3.01	2.97	2.93
	9	5.12	4.26	3.86	3.63	3.48	3.37	3.29	3.23	3.18	3.14	3.07	3.01	2.94	2.90	2.86	2.83	2.79	2.75	2.71
	10	4.96	4.10	3.71	3.48	3.33	3.22	3.14	3.07	3.02	2.98	2.91	2.85	2.77	2.74	2.70	2.66	2.62	2.58	2.54
	11	4.81	3.95	3.56	3.33	3.18	3.07	3.00	2.94	2.89	2.85	2.78	2.72	2.65	2.61	2.57	2.53	2.49	2.45	2.40
	12	4.75	3.89	3.49	3.26	3.11	3.00	2.91	2.85	2.80	2.75	2.69	2.62	2.55	2.51	2.47	2.43	2.38	2.34	2.30
	13	4.67	3.81	3.41	3.18	3.03	2.92	2.83	2.77	2.71	2.67	2.60	2.53	2.46	2.42	2.38	2.34	2.30	2.25	2.21
	14	4.60	3.74	3.34	3.11	2.96	2.85	2.76	2.70	2.65	2.60	2.53	2.46	2.39	2.35	2.31	2.27	2.22	2.18	2.13
	15	4.54	3.68	3.29	3.06	2.90	2.79	2.71	2.64	2.59	2.54	2.48	2.40	2.33	2.29	2.25	2.20	2.16	2.11	2.07
	16	4.49	3.63	3.24	3.01	2.85	2.74	2.66	2.59	2.54	2.49	2.42	2.35	2.28	2.24	2.19	2.15	2.11	2.06	2.01
	17	4.45	3.59	3.20	2.97	2.81	2.70	2.61	2.55	2.49	2.45	2.38	2.31	2.23	2.19	2.15	2.10	2.06	2.01	1.96
	18	4.41	3.55	3.16	2.93	2.77	2.66	2.58	2.51	2.46	2.41	2.34	2.27	2.19	2.15	2.11	2.06	2.02	1.97	1.92
	19	4.38	3.52	3.13	2.90	2.74	2.63	2.54	2.48	2.42	2.38	2.31	2.23	2.16	2.11	2.07	2.03	1.98	1.93	1.88
	20	4.35	3.49	3.10	2.87	2.71	2.60	2.51	2.45	2.39	2.35	2.28	2.20	2.12	2.08	2.04	1.99	1.95	1.90	1.84
	21	4.32	3.47	3.07	2.84	2.68	2.57	2.49	2.42	2.37	2.32	2.25	2.18	2.10	2.05	2.01	1.96	1.92	1.87	1.81
	22	4.30	3.44	3.05	2.82	2.66	2.55	2.46	2.40	2.34	2.30	2.23	2.15	2.07	2.03	1.98	1.94	1.89	1.84	1.78
	23	4.28	3.42	3.03	2.80	2.64	2.53	2.44	2.37	2.32	2.27	2.20	2.13	2.05	2.01	1.96	1.91	1.86	1.81	1.75
	24	4.26	3.40	3.01	2.78	2.62	2.51	2.42	2.36	2.30	2.25	2.18	2.11	2.03	1.98	1.94	1.89	1.84	1.79	1.73
	25	4.24	3.39	2.99	2.76	2.60	2.49	2.40	2.34	2.28	2.24	2.16	2.09	2.01	1.96	1.92	1.87	1.82	1.77	1.71
	30	4.17	3.32	2.92	2.69	2.53	2.42	2.33	2.27	2.21	2.16	2.09	2.01	1.93	1.89	1.84	1.79	1.74	1.68	1.62
	40	4.08	3.23	2.84	2.61	2.45	2.34	2.25	2.18	2.12	2.08	2.00	1.92	1.84	1.79	1.74	1.69	1.64	1.58	1.51
	60	4.00	3.15	2.76	2.53	2.37	2.25	2.17	2.10	2.04	1.99	1.92	1.84	1.75	1.70	1.65	1.59	1.53	1.47	1.39
	120	3.92	3.07	2.68	2.45	2.29	2.18	2.09	2.02	1.96	1.91	1.83	1.75	1.66	1.61	1.55	1.50	1.43	1.35	1.25
	∞	3.84	3.00	2.60	2.37	2.21	2.10	2.01	1.94	1.88	1.83	1.75	1.67	1.57	1.52	1.46	1.39	1.32	1.22	1.09

TABLE A-7b. F DISTRIBUTION, UPPER 1% POINTS ($F_{.01}$)

		Degrees of freedom for numerator																		
		1	2	3	4	5	6	7	8	9	10	12	15	20	24	30	40	60	120	∞
Degrees of freedom for denominator	1	4.052	5.000	5.403	5.625	5.764	5.859	5.928	5.982	6.023	6.056	6.106	6.157	6.209	6.235	6.261	6.287	6.313	6.339	6.365
	2	98.5	99.0	99.2	99.2	99.3	99.3	99.4	99.4	99.4	99.4	99.4	99.4	99.4	99.4	99.5	99.5	99.5	99.5	99.5
	3	31.1	30.8	29.5	28.7	28.2	27.9	27.7	27.5	27.3	27.2	27.1	26.9	26.7	26.6	26.5	26.4	26.3	26.2	26.1
	4	21.2	18.0	16.7	16.0	15.5	15.2	15.0	14.8	14.7	14.5	14.4	14.2	14.0	13.9	13.8	13.7	13.7	13.6	13.5
	5	16.3	13.3	12.1	11.4	11.0	10.7	10.5	10.3	10.2	10.1	9.89	9.72	9.55	9.47	9.38	9.29	9.20	9.11	9.02
	6	13.7	10.9	9.78	9.1	8.75	8.47	8.26	8.10	7.98	7.87	7.72	7.56	7.40	7.31	7.23	7.14	7.06	6.97	6.88
	7	12.2	9.55	8.45	7.8	7.46	7.19	6.99	6.81	6.72	6.62	6.47	6.31	6.16	6.07	5.99	5.91	5.82	5.74	5.65
	8	11.3	8.65	7.59	7.0	6.63	6.37	6.18	6.03	5.91	5.81	5.67	5.52	5.36	5.28	5.20	5.12	5.03	4.95	4.86
	9	10.6	8.02	6.99	6.42	6.06	5.80	5.61	5.47	5.35	5.25	5.11	4.96	4.81	4.73	4.65	4.57	4.48	4.40	4.31
	10	10.0	7.36	6.35	5.79	5.44	5.19	5.00	4.85	4.71	4.59	4.44	4.29	4.14	4.06	3.98	3.90	3.81	3.73	3.64
	11	9.65	7.21	6.22	5.67	5.32	5.07	4.89	4.74	4.63	4.51	4.40	4.25	4.10	4.02	3.94	3.86	3.78	3.69	3.61
	12	9.33	6.93	5.95	5.41	5.06	4.82	4.64	4.50	4.39	4.30	4.16	4.01	3.86	3.78	3.70	3.62	3.54	3.45	3.37
	13	9.07	6.70	5.74	5.21	4.86	4.62	4.44	4.30	4.19	4.10	3.96	3.81	3.66	3.58	3.50	3.42	3.34	3.25	3.17
	14	8.86	6.51	5.56	5.03	4.70	4.46	4.28	4.14	4.03	3.94	3.80	3.65	3.51	3.43	3.35	3.27	3.18	3.09	3.01
	15	8.68	6.36	5.42	4.89	4.56	4.32	4.14	4.00	3.89	3.80	3.67	3.52	3.37	3.29	3.21	3.13	3.04	2.95	2.87
	16	8.53	6.23	5.29	4.77	4.44	4.20	4.03	3.89	3.78	3.69	3.55	3.41	3.26	3.18	3.10	3.02	2.93	2.84	2.75
	17	8.40	6.11	5.19	4.67	4.34	4.10	3.93	3.79	3.68	3.59	3.46	3.31	3.16	3.08	3.00	2.92	2.83	2.74	2.65
	18	8.29	6.01	5.09	4.58	4.25	4.01	3.84	3.71	3.60	3.51	3.37	3.23	3.08	3.00	2.92	2.84	2.75	2.66	2.57
	19	8.19	5.93	5.01	4.50	4.17	3.93	3.77	3.63	3.52	3.43	3.30	3.15	3.00	2.92	2.84	2.76	2.67	2.58	2.49
	20	8.10	5.85	4.91	4.43	4.10	3.87	3.70	3.56	3.46	3.37	3.23	3.09	2.94	2.86	2.78	2.69	2.61	2.52	2.42
	21	8.02	5.78	4.87	4.37	4.04	3.81	3.64	3.51	3.40	3.31	3.17	3.03	2.88	2.80	2.72	2.64	2.55	2.46	2.36
	22	7.95	5.72	4.82	4.31	3.99	3.76	3.59	3.45	3.35	3.26	3.12	2.98	2.83	2.75	2.67	2.58	2.50	2.40	2.31
	23	7.88	5.66	4.76	4.26	3.94	3.71	3.54	3.41	3.30	3.21	3.07	2.93	2.78	2.70	2.62	2.54	2.45	2.35	2.26
	24	7.82	5.61	4.72	4.22	3.90	3.67	3.50	3.36	3.26	3.17	3.03	2.89	2.74	2.66	2.58	2.49	2.40	2.31	2.21
	25	7.77	5.57	4.68	4.18	3.86	3.63	3.46	3.32	3.22	3.13	2.99	2.85	2.70	2.62	2.53	2.45	2.36	2.27	2.17
30	7.56	5.39	4.51	4.02	3.70	3.47	3.30	3.17	3.07	2.98	2.84	2.70	2.55	2.47	2.39	2.30	2.21	2.11	2.01	
40	7.31	5.18	4.31	3.83	3.51	3.29	3.12	2.99	2.89	2.80	2.66	2.52	2.37	2.29	2.21	2.12	2.03	1.93	1.83	
60	7.08	4.98	4.13	3.65	3.33	3.12	2.95	2.82	2.72	2.63	2.50	2.35	2.20	2.12	2.04	1.95	1.86	1.76	1.66	
120	6.85	4.79	3.95	3.48	3.17	2.96	2.79	2.66	2.56	2.47	2.34	2.19	2.03	1.95	1.86	1.76	1.66	1.56	1.46	
∞	6.63	4.61	3.78	3.32	3.02	2.80	2.64	2.51	2.41	2.32	2.18	2.04	1.88	1.79	1.70	1.59	1.47	1.32	1.10	

TABLE A-4

TABLE A-17. CRITICAL VALUES FOR COCHRAN'S TEST*

Values given are for the statistic $(\text{largest } s^2)/(\sum s^2)$, where each of the k values of s^2 has ν degrees of freedom.

Percentile 95

$\nu \backslash k$	1	2	3	4	5	6	7	8	9	10	16	36	144	∞
2	0.9985	0.9750	0.9492	0.9057	0.8772	0.8534	0.8332	0.8159	0.8010	0.7880	0.7341	0.6602	0.5813	0.5090
3	0.9609	0.8709	0.7977	0.7457	0.7071	0.6771	0.6530	0.6333	0.6167	0.6027	0.5466	0.4748	0.4031	0.3373
4	0.9063	0.7679	0.6841	0.6287	0.5895	0.5598	0.5365	0.5175	0.5017	0.4881	0.4309	0.3720	0.3093	0.2560
5	0.8412	0.6838	0.5981	0.5441	0.5065	0.4783	0.4564	0.4387	0.4241	0.4118	0.3615	0.3066	0.2513	0.2048
6	0.7898	0.6161	0.5321	0.4804	0.4447	0.4184	0.3980	0.3817	0.3682	0.3568	0.3133	0.2612	0.2119	0.1717
7	0.7271	0.5612	0.4809	0.4307	0.3974	0.3726	0.3535	0.3381	0.3259	0.3151	0.2756	0.2278	0.1833	0.1429
8	0.6798	0.5157	0.4377	0.3910	0.3595	0.3362	0.3185	0.3043	0.2926	0.2829	0.2462	0.2022	0.1616	0.1250
9	0.6383	0.4775	0.4027	0.3584	0.3286	0.3067	0.2901	0.2768	0.2659	0.2568	0.2226	0.1820	0.1446	0.1111
10	0.6020	0.4459	0.3733	0.3311	0.3024	0.2823	0.2660	0.2541	0.2439	0.2353	0.2032	0.1655	0.1308	0.1000
12	0.5410	0.3924	0.3264	0.2880	0.2621	0.2439	0.2299	0.2187	0.2098	0.2020	0.1737	0.1403	0.1100	0.0843
15	0.4709	0.3346	0.2758	0.2419	0.2195	0.2034	0.1911	0.1815	0.1736	0.1671	0.1429	0.1144	0.0889	0.0667
20	0.3891	0.2705	0.2265	0.1929	0.1735	0.1602	0.1501	0.1422	0.1357	0.1303	0.1108	0.0879	0.0675	0.0500
24	0.3434	0.2351	0.1907	0.1656	0.1493	0.1374	0.1286	0.1216	0.1160	0.1113	0.0942	0.0743	0.0567	0.0417
30	0.2929	0.1980	0.1593	0.1377	0.1247	0.1137	0.1061	0.1002	0.0958	0.0921	0.0771	0.0601	0.0457	0.0333
40	0.2370	0.1576	0.1259	0.1082	0.0968	0.0887	0.0827	0.0780	0.0745	0.0713	0.0595	0.0492	0.0377	0.0283
60	0.1737	0.1131	0.0895	0.0765	0.0682	0.0623	0.0583	0.0552	0.0520	0.0497	0.0411	0.0346	0.0281	0.0167
120	0.0998	0.0632	0.0495	0.0419	0.0371	0.0337	0.0312	0.0292	0.0279	0.0266	0.0218	0.0165	0.0120	0.0083
∞	0	0	0	0	0	0	0	0	0	0	0	0	0	0

* Reproduced with permission from C. Eisenhart, M. W. Hastay, W. A. Wallis, *Techniques of Statistical Analysis*, chap. 15. McGraw-Hill Book Company, Inc., New York, 1947.

TABLE A-17. CRITICAL VALUES FOR COCHRAN'S TEST (Continued)

Percentile 99

$\nu \backslash k$	1	2	3	4	5	6	7	8	9	10	16	36	144	∞
2	0.9999	0.9950	0.9854	0.9686	0.9573	0.9472	0.9388	0.9323	0.9274	0.9239	0.7949	0.7067	0.6032	0.5090
3	0.9933	0.9433	0.8933	0.8333	0.7933	0.7609	0.7333	0.7107	0.6912	0.6743	0.6059	0.5153	0.4239	0.3373
4	0.9676	0.8633	0.7833	0.7212	0.6761	0.6410	0.6129	0.5897	0.5702	0.5536	0.4881	0.4057	0.3231	0.2560
5	0.9279	0.7885	0.7077	0.6329	0.5875	0.5531	0.5259	0.5037	0.4854	0.4697	0.4094	0.3351	0.2615	0.2020
6	0.8828	0.7218	0.6411	0.5635	0.5195	0.4866	0.4608	0.4401	0.4229	0.4081	0.3529	0.2858	0.2229	0.1667
7	0.8376	0.6644	0.5837	0.5080	0.4659	0.4347	0.4105	0.3911	0.3751	0.3616	0.3105	0.2494	0.1929	0.1429
8	0.7945	0.6152	0.5345	0.4627	0.4226	0.3932	0.3704	0.3522	0.3373	0.3248	0.2779	0.2214	0.1709	0.1250
9	0.7541	0.5727	0.4919	0.4251	0.3870	0.3592	0.3378	0.3207	0.3067	0.2943	0.2514	0.1992	0.1527	0.1111
10	0.7175	0.5358	0.4550	0.3934	0.3572	0.3308	0.3106	0.2945	0.2813	0.2703	0.2297	0.1811	0.1376	0.1000
12	0.6528	0.4751	0.3949	0.3428	0.3099	0.2861	0.2680	0.2535	0.2419	0.2320	0.1961	0.1533	0.1157	0.0843
15	0.5747	0.3969	0.3317	0.2882	0.2593	0.2386	0.2228	0.2104	0.2002	0.1918	0.1632	0.1264	0.0931	0.0667
20	0.4799	0.3207	0.2554	0.2288	0.2048	0.1877	0.1748	0.1646	0.1567	0.1501	0.1248	0.0969	0.0699	0.0500
24	0.4247	0.2871	0.2295	0.1970	0.1759	0.1608	0.1495	0.1406	0.1338	0.1283	0.1060	0.0810	0.0595	0.0417
30	0.3642	0.2412	0.1913	0.1635	0.1451	0.1327	0.1232	0.1157	0.1100	0.1054	0.0867	0.0658	0.0489	0.0333
40	0.2949	0.1915	0.1508	0.1281	0.1133	0.1033	0.0957	0.0898	0.0853	0.0816	0.0668	0.0503	0.0363	0.0266
60	0.2151	0.1371	0.1099	0.0902	0.0796	0.0722	0.0668	0.0625	0.0591	0.0567	0.0461	0.0344	0.0245	0.0167
120	0.1225	0.0759	0.0585	0.0489	0.0429	0.0387	0.0357	0.0334	0.0316	0.0302	0.0242	0.0178	0.0129	0.0083
∞	0	0	0	0	0	0	0	0	0	0	0	0	0	0

TABLE J
Percentage Points of the F Distribution*

TABLE J—Continued

ν_1	P	$\nu_1 = 1$	2	3	4	5	6	7	8	9	10	12	15	20	24	30	60	120	∞
0.500	0.500	1.00	1.50	1.71	1.82	1.89	1.94	1.98	2.00	2.03	2.04	2.07	2.09	2.12	2.13	2.15	2.17	2.18	2.20
0.100	0.100	39.9	49.5	53.6	55.8	57.2	58.2	58.9	59.4	59.9	60.2	60.7	61.2	61.7	62.0	62.3	62.8	63.1	63.3
0.050	0.050	161	200	216	225	230	234	237	239	241	242	244	246	248	250	252	254	255	254
1	0.025	648	800	864	900	922	937	948	957	963	969	977	983	987	990	991	992	993	994
0.010	0.010	4,050	5,000	5,400	5,620	5,760	5,860	5,930	5,980	6,020	6,060	6,110	6,160	6,210	6,235	6,260	6,310	6,340	6,370
0.005	0.005	16,200	20,000	21,600	22,300	23,100	23,400	23,700	23,900	24,100	24,200	24,400	24,600	24,800	24,900	25,000	25,300	25,400	25,500
0.001	0.001	405,284	500,000	540,379	562,500	576,405	585,937	598,144				610,667			623,497				636,619
0.500	0.500	0.667	1.00	1.13	1.21	1.25	1.28	1.30	1.32	1.33	1.34	1.36	1.38	1.39	1.40	1.41	1.43	1.43	1.44
0.100	0.100	8.53	9.00	9.16	9.24	9.29	9.33	9.35	9.37	9.38	9.39	9.41	9.42	9.44	9.45	9.46	9.47	9.48	9.49
0.050	0.050	18.5	19.0	19.2	19.2	19.3	19.3	19.4	19.4	19.4	19.4	19.4	19.4	19.4	19.5	19.5	19.5	19.5	19.5
2	0.025	38.5	39.0	39.2	39.2	39.3	39.3	39.4	39.4	39.4	39.4	39.4	39.4	39.4	39.5	39.5	39.5	39.5	39.5
0.010	0.010	98.5	99.0	99.2	99.2	99.3	99.3	99.4	99.4	99.4	99.4	99.4	99.4	99.4	99.5	99.5	99.5	99.5	99.5
0.005	0.005	199	199	199	199	199	199	199	199	199	199	199	199	199	199	199	199	199	200
0.001	0.001	998.5	999.0	999.2	999.2	999.3	999.3	999.4	999.4			999.4			999.5				999.5
0.500	0.500	0.585	0.881	1.00	1.06	1.10	1.13	1.15	1.16	1.17	1.18	1.20	1.21	1.23	1.23	1.24	1.25	1.26	1.27
0.100	0.100	5.54	5.46	5.39	5.34	5.31	5.28	5.27	5.27	5.24	5.23	5.22	5.20	5.18	5.18	5.17	5.15	5.14	5.13
0.050	0.050	10.1	9.55	9.28	9.12	9.01	8.94	8.89	8.85	8.81	8.79	8.74	8.70	8.66	8.64	8.62	8.57	8.55	8.53
3	0.025	17.4	16.0	15.4	15.1	14.9	14.7	14.6	14.5	14.5	14.4	14.3	14.3	14.2	14.1	14.1	14.0	13.9	13.9
0.010	0.010	34.1	30.8	29.5	28.7	28.2	27.9	27.7	27.5	27.3	27.2	27.1	26.9	26.7	26.6	26.5	26.3	26.2	26.1
0.005	0.005	55.6	49.8	47.5	46.2	45.4	44.8	44.4	44.1	43.9	43.7	43.4	43.1	42.8	42.6	42.5	42.1	42.0	41.8
0.001	0.001	167.5	148.5	141.1	137.1	134.6	132.8		130.6			128.3			125.9				123.5
0.500	0.500	0.549	0.828	0.941	1.00	1.04	1.06	1.08	1.09	1.10	1.11	1.13	1.14	1.15	1.16	1.16	1.18	1.18	1.19
0.100	0.100	4.54	4.32	4.19	4.11	4.05	4.01	3.98	3.95	3.94	3.92	3.90	3.87	3.84	3.83	3.82	3.79	3.78	3.76
0.050	0.050	7.71	6.94	6.59	6.39	6.26	6.16	6.09	6.04	6.00	5.96	5.91	5.86	5.80	5.77	5.75	5.69	5.66	5.63
4	0.025	12.2	10.6	9.98	9.60	9.36	9.20	9.07	8.98	8.90	8.81	8.75	8.66	8.56	8.51	8.46	8.36	8.31	8.26
0.010	0.010	21.2	18.0	16.7	16.0	15.5	15.2	15.0	14.8	14.7	14.5	14.4	14.2	14.0	13.9	13.8	13.7	13.6	13.5
0.005	0.005	31.3	26.3	24.3	23.2	22.5	22.0	21.6	21.4	21.1	21.0	20.7	20.4	20.2	20.0	19.9	19.6	19.5	19.3
0.001	0.001	74.1	61.3	56.2	53.4	51.7	50.5		49.0			47.4			45.8				44.1
0.500	0.500	0.528	0.799	0.907	0.965	1.00	1.02	1.04	1.05	1.06	1.07	1.09	1.10	1.11	1.12	1.12	1.14	1.14	1.15
0.100	0.100	3.78	3.46	3.32	3.22	3.15	3.10	3.07	3.04	3.02	3.00	3.00	2.97	2.94	2.93	2.92	2.89	2.88	2.87
0.050	0.050	6.61	5.79	5.41	5.19	5.05	4.95	4.88	4.82	4.77	4.74	4.68	4.62	4.56	4.56	4.53	4.43	4.40	4.37
5	0.025	10.0	8.43	7.76	7.39	7.15	6.98	6.85	6.76	6.68	6.62	6.52	6.43	6.33	6.28	6.23	6.12	6.07	6.02
0.010	0.010	16.3	13.3	12.1	11.4	11.0	10.7	10.5	10.3	10.2	10.1	9.89	9.72	9.55	9.47	9.38	9.20	9.11	9.02
0.005	0.005	22.8	18.3	16.5	15.6	14.9	14.5	14.2	14.0	13.8	13.6	13.4	13.1	12.9	12.8	12.7	12.4	12.3	12.1
0.001	0.001	47.0	36.6	33.2	31.1	29.8	28.8		27.6			26.4			25.1				23.8
0.500	0.500	0.515	0.780	0.886	0.942	0.977	1.00	1.02	1.03	1.04	1.05	1.06	1.07	1.08	1.09	1.10	1.11	1.12	1.12
0.100	0.100	3.38	3.06	2.92	2.82	2.75	2.70	2.67	2.65	2.64	2.63	2.62	2.60	2.58	2.57	2.56	2.54	2.53	2.52
0.050	0.050	5.99	5.14	4.76	4.53	4.39	4.28	4.21	4.15	4.10	4.06	4.00	3.94	3.87	3.84	3.81	3.74	3.70	3.67
6	0.025	8.81	7.26	6.60	6.23	5.99	5.82	5.70	5.60	5.52	5.46	5.37	5.27	5.17	5.12	5.07	4.96	4.90	4.85
0.010	0.010	13.7	10.9	9.78	9.15	8.75	8.47	8.26	8.10	7.98	7.87	7.72	7.56	7.40	7.31	7.23	7.06	6.97	6.88
0.005	0.005	18.6	14.5	12.9	12.0	11.5	11.1	10.8	10.6	10.4	10.2	10.0	9.81	9.59	9.47	9.36	9.12	9.00	8.88
0.001	0.001	35.5	27.0	23.7	21.9	20.8	20.0		19.0			18.0			16.9				15.8
0.500	0.500	0.506	0.767	0.871	0.926	0.960	0.983	1.00	1.01	1.02	1.03	1.04	1.05	1.07	1.07	1.08	1.09	1.10	1.10
0.100	0.100	3.59	3.26	3.07	2.96	2.88	2.83	2.78	2.75	2.72	2.70	2.67	2.63	2.59	2.58	2.56	2.51	2.49	2.47
0.050	0.050	5.99	4.74	4.35	4.12	3.97	3.87	3.79	3.73	3.68	3.64	3.57	3.51	3.44	3.41	3.38	3.30	3.27	3.23
7	0.025	8.07	6.54	5.89	5.52	5.29	5.12	4.99	4.90	4.82	4.76	4.67	4.57	4.47	4.42	4.36	4.25	4.20	4.14
0.010	0.010	12.2	9.53	8.45	7.85	7.46	7.19	6.99	6.84	6.72	6.62	6.47	6.31	6.16	6.07	5.99	5.82	5.74	5.65
0.005	0.005	16.2	12.4	10.9	10.1	9.52	9.16	8.89	8.68	8.51	8.38	8.18	7.97	7.75	7.65	7.53	7.31	7.19	7.08
0.001	0.001	29.2	21.7	18.8	17.2	16.2	15.5		14.6			13.7			12.7				11.7

* The 0.001 points of Table J are abridged with permission from Table V of R. A. Fisher and F. Yates, *Statistical Tables for Biological, Agricultural and Medical Research*, Oliver & Boyd Ltd. (1951). The rest of the table is abridged with permission from *Tables for Inverting the F Distribution*, by M. M. R. Thompson, *Biometrika*, Vol. XXXIII (1944-46) pp. 73-84.

TABLE J-Continued

TABLE J-Continued

P	$P_1 = 1$	2	3	4	5	6	7	8	9	10	12	15	20	24	30	60	120	∞
0.500	0.499	0.757	0.860	0.915	0.948	0.971	0.988	1.00	1.01	1.02	1.03	1.04	1.05	1.06	1.07	1.08	1.08	1.09
0.100	0.346	3.11	2.92	2.81	2.73	2.67	2.62	2.59	2.56	2.54	2.50	2.46	2.42	2.40	2.38	2.34	2.32	2.29
0.050	5.32	4.46	4.07	3.84	3.69	3.58	3.50	3.44	3.39	3.35	3.28	3.22	3.15	3.12	3.08	3.01	2.97	2.93
0.025	7.57	6.06	5.42	5.05	4.82	4.65	4.53	4.43	4.36	4.30	4.20	4.10	4.00	3.95	3.89	3.78	3.73	3.67
0.010	11.3	8.65	7.59	7.01	6.63	6.37	6.18	6.03	5.91	5.81	5.67	5.52	5.36	5.28	5.20	5.03	4.95	4.86
0.005	14.7	11.0	9.60	8.81	8.30	7.95	7.69	7.50	7.34	7.21	7.01	6.81	6.61	6.50	6.40	6.18	6.06	5.95
0.001	25.4	18.5	15.8	14.4	13.5	12.9	12.3	12.0	11.7	11.2	10.7	10.2	9.81	9.57	9.34	9.06	8.81	8.57
0.500	0.494	0.749	0.852	0.906	0.939	0.962	0.978	0.990	1.00	1.01	1.02	1.03	1.04	1.05	1.05	1.07	1.07	1.08
0.100	3.36	3.01	2.81	2.69	2.61	2.55	2.51	2.47	2.44	2.42	2.38	2.34	2.30	2.28	2.25	2.21	2.18	2.16
0.050	5.12	4.26	3.86	3.63	3.48	3.37	3.29	3.23	3.18	3.14	3.07	3.01	2.94	2.90	2.86	2.79	2.75	2.71
0.025	7.21	5.71	5.08	4.72	4.48	4.32	4.20	4.10	4.03	3.96	3.87	3.77	3.67	3.61	3.56	3.45	3.39	3.33
0.010	10.6	8.02	6.99	6.42	6.06	5.80	5.61	5.47	5.35	5.26	5.11	4.96	4.81	4.73	4.65	4.48	4.40	4.31
0.005	13.6	10.1	8.72	7.96	7.47	7.13	6.88	6.69	6.54	6.42	6.23	6.03	5.83	5.73	5.62	5.41	5.30	5.19
0.001	22.9	16.4	13.9	12.6	11.7	11.1	10.4	10.4	10.1	9.85	9.57	9.27	8.97	8.72	8.48	8.18	7.91	7.61
0.500	0.490	0.743	0.845	0.899	0.932	0.954	0.971	0.983	0.992	1.00	1.01	1.02	1.03	1.04	1.05	1.06	1.06	1.07
0.100	3.29	2.92	2.73	2.61	2.52	2.46	2.41	2.38	2.35	2.32	2.28	2.24	2.20	2.18	2.16	2.11	2.08	2.06
0.050	4.96	4.10	3.71	3.48	3.33	3.22	3.14	3.07	3.02	2.98	2.91	2.84	2.77	2.74	2.70	2.62	2.58	2.54
0.025	6.94	5.46	4.83	4.47	4.24	4.07	3.95	3.85	3.78	3.72	3.62	3.52	3.42	3.37	3.31	3.20	3.14	3.08
0.010	10.0	7.56	6.55	5.99	5.64	5.39	5.20	5.06	4.94	4.85	4.71	4.56	4.41	4.33	4.25	4.08	4.00	3.91
0.005	12.8	9.43	8.08	7.34	6.87	6.54	6.30	6.12	5.97	5.85	5.66	5.47	5.27	5.17	5.07	4.86	4.75	4.64
0.001	21.0	14.9	12.6	11.3	10.5	9.92	9.20	9.20	8.85	8.45	8.05	7.65	7.25	7.00	6.75	6.45	6.15	5.85
0.500	0.484	0.735	0.835	0.888	0.921	0.943	0.959	0.972	0.981	0.989	1.00	1.01	1.02	1.03	1.03	1.05	1.05	1.06
0.100	3.18	2.81	2.61	2.48	2.39	2.33	2.28	2.24	2.21	2.19	2.15	2.10	2.06	2.04	2.01	1.96	1.93	1.90
0.050	4.75	3.89	3.49	3.26	3.11	3.00	2.91	2.85	2.80	2.75	2.69	2.62	2.54	2.51	2.47	2.38	2.34	2.30
0.025	6.55	5.10	4.47	4.12	3.89	3.73	3.61	3.51	3.43	3.37	3.28	3.18	3.07	3.02	2.96	2.85	2.79	2.72
0.010	9.33	6.93	5.95	5.41	5.06	4.82	4.64	4.50	4.39	4.30	4.16	4.01	3.86	3.78	3.70	3.54	3.45	3.36
0.005	11.8	8.51	7.23	6.52	6.07	5.76	5.52	5.35	5.20	5.09	4.91	4.72	4.53	4.43	4.33	4.12	4.01	3.90
0.001	18.6	13.0	10.8	9.63	8.89	8.38	7.71	7.71	7.35	6.95	6.55	6.15	5.75	5.50	5.25	4.95	4.65	4.35
0.500	0.478	0.726	0.826	0.878	0.911	0.933	0.949	0.960	0.970	0.977	0.989	1.00	1.01	1.02	1.02	1.03	1.04	1.05
0.100	3.07	2.70	2.49	2.36	2.27	2.21	2.16	2.12	2.09	2.06	2.02	1.97	1.92	1.90	1.87	1.82	1.79	1.76
0.050	4.54	3.68	3.29	3.06	2.90	2.79	2.71	2.64	2.59	2.54	2.48	2.40	2.33	2.29	2.25	2.16	2.11	2.07
0.025	6.20	4.77	4.15	3.80	3.58	3.41	3.29	3.20	3.12	3.06	2.96	2.86	2.76	2.70	2.64	2.52	2.46	2.40
0.010	8.68	6.36	5.42	4.89	4.56	4.32	4.14	4.00	3.89	3.80	3.67	3.52	3.37	3.29	3.21	3.05	2.96	2.87
0.005	10.8	7.70	6.48	5.80	5.37	5.07	4.85	4.67	4.54	4.42	4.25	4.07	3.88	3.79	3.69	3.48	3.37	3.26
0.001	16.6	11.34	9.34	8.25	7.57	7.09	6.47	6.47	6.11	5.71	5.31	4.91	4.51	4.26	4.01	3.71	3.41	3.11
0.500	0.472	0.718	0.816	0.868	0.900	0.922	0.938	0.950	0.959	0.966	0.977	0.989	1.00	1.01	1.01	1.02	1.03	1.03
0.100	2.97	2.59	2.38	2.25	2.16	2.09	2.04	2.00	1.96	1.94	1.89	1.84	1.79	1.77	1.74	1.68	1.64	1.61
0.050	4.35	3.49	3.10	2.87	2.71	2.60	2.51	2.45	2.39	2.35	2.28	2.20	2.12	2.08	2.04	1.95	1.90	1.84
0.025	5.87	4.46	3.86	3.51	3.29	3.13	3.01	2.91	2.84	2.77	2.68	2.57	2.46	2.41	2.35	2.22	2.16	2.09
0.010	8.10	5.85	4.94	4.43	4.10	3.87	3.70	3.56	3.46	3.37	3.23	3.09	2.94	2.86	2.78	2.61	2.52	2.42
0.005	9.94	6.99	5.82	5.17	4.76	4.47	4.26	4.09	3.96	3.85	3.68	3.50	3.32	3.22	3.12	2.92	2.81	2.69
0.001	14.8	9.95	8.10	7.10	6.46	6.02	5.44	5.44	5.08	4.68	4.28	3.88	3.48	3.23	3.03	2.73	2.43	2.13
0.500	0.469	0.714	0.812	0.863	0.895	0.917	0.932	0.944	0.953	0.961	0.972	0.983	0.994	1.00	1.01	1.02	1.02	1.03
0.100	2.93	2.54	2.33	2.19	2.10	2.04	1.98	1.94	1.91	1.88	1.83	1.78	1.73	1.70	1.67	1.61	1.57	1.53
0.050	4.26	3.40	3.01	2.78	2.62	2.51	2.42	2.36	2.30	2.25	2.18	2.11	2.03	1.98	1.94	1.84	1.79	1.73
0.025	5.72	4.32	3.72	3.38	3.15	2.99	2.87	2.78	2.70	2.64	2.54	2.44	2.33	2.27	2.21	2.08	2.01	1.94
0.010	7.82	5.61	4.72	4.22	3.90	3.67	3.50	3.36	3.26	3.17	3.03	2.89	2.74	2.66	2.58	2.40	2.31	2.21
0.005	9.55	6.66	5.52	4.89	4.49	4.20	3.99	3.83	3.69	3.59	3.43	3.25	3.06	2.97	2.87	2.66	2.55	2.43
0.001	14.0	9.34	7.55	6.59	5.98	5.55	4.99	4.99	4.63	4.23	3.83	3.43	3.03	2.78	2.58	2.28	2.03	1.73

TABLE J - Continued

TABLE J - Concluded

P_1	P	$P_1 = 1$	2	3	4	5	6	7	8	9	10	12	15	20	24	30	60	120	∞
0.500	0.466	0.709	0.807	0.858	0.890	0.912	0.927	0.939	0.948	0.955	0.966	0.978	0.989	0.994	0.994	1.00	1.01	1.02	1.02
0.100	2.88	2.49	2.28	2.14	2.05	1.98	1.93	1.88	1.85	1.82	1.77	1.72	1.67	1.64	1.61	1.54	1.54	1.50	1.46
0.050	4.17	3.32	2.92	2.69	2.53	2.42	2.33	2.27	2.21	2.16	2.09	2.01	1.93	1.86	1.81	1.74	1.74	1.68	1.62
0.025	5.57	4.18	3.59	3.25	3.03	2.87	2.75	2.65	2.57	2.51	2.41	2.31	2.20	2.14	2.07	1.94	1.94	1.87	1.79
0.010	7.56	5.39	4.31	4.02	3.70	3.47	3.30	3.17	3.07	2.98	2.84	2.70	2.55	2.47	2.39	2.21	2.21	2.11	2.01
0.005	9.18	6.35	5.24	4.62	4.23	3.95	3.74	3.58	3.45	3.34	3.18	3.01	2.82	2.73	2.63	2.42	2.42	2.30	2.18
0.001	13.29	8.77	7.05	6.12	5.53	5.12	4.88	4.58	4.45	4.00	3.31	2.74	2.57	2.39	2.29	2.19	1.96	1.83	1.69
0.500	0.461	0.701	0.798	0.849	0.880	0.901	0.917	0.928	0.937	0.945	0.956	0.967	0.978	0.983	0.989	0.989	1.00	1.01	1.01
0.100	2.79	2.39	2.18	2.04	1.95	1.87	1.82	1.77	1.74	1.71	1.66	1.60	1.54	1.51	1.48	1.40	1.40	1.35	1.29
0.050	4.00	3.15	2.76	2.53	2.37	2.25	2.17	2.10	2.04	1.99	1.92	1.84	1.75	1.70	1.65	1.53	1.53	1.47	1.39
0.025	5.29	3.93	3.34	3.01	2.79	2.63	2.51	2.41	2.33	2.27	2.17	2.06	1.94	1.88	1.82	1.67	1.67	1.58	1.48
0.010	7.08	4.98	4.13	3.65	3.34	3.12	2.95	2.82	2.72	2.63	2.50	2.35	2.20	2.12	2.03	1.81	1.81	1.73	1.60
0.005	8.49	5.80	4.73	4.14	3.76	3.49	3.29	3.13	3.01	2.90	2.74	2.57	2.39	2.29	2.19	1.96	1.96	1.83	1.69
0.001	11.97	7.76	6.17	5.31	4.76	4.37	4.07	3.87	3.74	3.31	2.74	2.57	2.39	2.29	2.19	1.96	1.83	1.73	1.60
0.500	0.458	0.697	0.793	0.844	0.875	0.896	0.912	0.923	0.932	0.939	0.950	0.961	0.972	0.978	0.983	0.983	0.994	1.00	1.01
0.100	2.75	2.35	2.13	1.99	1.90	1.82	1.77	1.72	1.68	1.65	1.60	1.55	1.48	1.45	1.41	1.32	1.32	1.26	1.19
0.050	3.92	3.07	2.68	2.45	2.29	2.18	2.09	2.02	1.96	1.91	1.83	1.75	1.66	1.61	1.55	1.43	1.43	1.35	1.25
0.025	5.15	3.80	3.23	2.89	2.67	2.52	2.39	2.30	2.22	2.16	2.05	1.95	1.82	1.76	1.69	1.53	1.53	1.43	1.31
0.010	6.85	4.79	3.95	3.48	3.17	2.96	2.79	2.66	2.56	2.47	2.34	2.19	2.03	1.95	1.86	1.66	1.66	1.53	1.38
0.005	8.18	5.54	4.50	3.92	3.55	3.28	3.09	2.93	2.81	2.71	2.54	2.37	2.19	2.09	1.98	1.75	1.75	1.61	1.43
0.001	11.38	7.31	5.79	4.95	4.42	4.04	3.74	3.55	3.42	3.02	2.45	2.37	2.19	2.09	1.98	1.75	1.61	1.53	1.36
0.500	0.455	0.693	0.789	0.839	0.870	0.891	0.907	0.918	0.927	0.934	0.945	0.956	0.967	0.972	0.978	0.989	0.989	0.994	1.00
0.100	2.71	2.30	2.08	1.94	1.85	1.77	1.72	1.67	1.63	1.60	1.55	1.49	1.42	1.38	1.34	1.24	1.24	1.17	1.00
0.050	3.84	3.00	2.60	2.37	2.21	2.10	2.01	1.94	1.88	1.83	1.75	1.67	1.57	1.52	1.46	1.32	1.32	1.22	1.00
0.025	5.02	3.69	3.12	2.79	2.57	2.41	2.29	2.19	2.11	2.05	1.94	1.83	1.71	1.64	1.57	1.39	1.39	1.27	1.00
0.010	6.63	4.61	3.78	3.32	3.02	2.80	2.64	2.51	2.41	2.32	2.18	2.04	1.88	1.79	1.70	1.47	1.47	1.32	1.00
0.005	7.88	5.30	4.28	3.72	3.35	3.09	2.90	2.74	2.62	2.52	2.36	2.19	2.00	1.90	1.79	1.53	1.53	1.36	1.00
0.001	10.8	6.91	5.42	4.62	4.10	3.74	3.42	3.27	3.14	2.82	2.24	2.19	2.00	1.90	1.79	1.53	1.36	1.36	1.00

APPENDIX B

Sample Forms and Instructions for Participants in
Round Robin

ARRADCOM - Product Assurance Directorate

Subject: Comparison of Test Methods for Determination of RDX in Composition B.

TO: Participants in Round Robin No. 1

1. Inclosed herewith are the following materials for subject Round Robin.

- a. Three samples of Comp B (A, B, C)
- b. Description of test methods
- c. Instructions for collecting and reporting data
- d. Two data sheets

2. The completed data sheets should be returned by 1 Jan 82.

3. The code number for your laboratory is .

Very truly yours,

ERIC R. BIXON
ARRADCOM
Product Assurance Directorate
Building 62
Dover, NJ 07801

It is important that all participants in the Round Robin receive the test procedures prior to receiving the samples and that all samples be received by the participants within a five (5) day period. It is extremely important that storage conditions for samples (prior to testing) be identical.

Individual testing by each laboratory should be completed within a seven (7) day period of receiving samples. Note ambient conditions in storage areas and laboratory areas.

ROUND ROBIN NO. 1

Sample Data Sheet for a Given Method

g of RDX in a 10g Sample

<u>LOT A</u>	<u>LOT B</u>	<u>LOT C</u>	<u>REPLICATE NO.</u>
-	-	-	1
-	-	-	2
-	-	-	3
-	-	-	4
-	-	-	5
-	-	-	6
-	-	-	7
-	-	-	8
-	-	-	9
-	-	-	10

Average Test Time, Minutes/Sample

Working Time for 10 Analyses _____

Total Elapsed Time for 10 Analyses _____

Laboratory Code No. _____

Method Used _____

Submitted by _____

Phone No. _____

Comments on Method

APPENDIX C

Data for Repeatability and Reproducibility Calculations

DATA FOR LOT A METHOD 1

LAB A	LAB B	LAB C	LAB D	LAB E
5.7760	5.7550	5.8270	5.7690	5.7760
5.7300	5.7580	5.7780	5.7870	5.8400
5.7350	5.8800	5.7120	5.7460	5.7730
5.7920	5.6840	5.7760	5.7770	5.7690
5.7180	5.7620	5.7090	5.8120	5.7710
5.7140	5.6720	5.7570	5.7800	5.7750
5.7760	5.7850	5.7590	5.7470	5.6980
5.6890	5.8170	5.7430	5.6550	5.7070
5.7710	5.7990	5.6970	5.7450	5.7950
5.7510	5.7930	5.7460	5.7980	5.8300

AVERAGES FOR EACH LAB

5.7452	5.7705	5.7504	5.7616	5.7734
--------	--------	--------	--------	--------

STANDARD DEVIATIONS FOR EACH LAB

.0333	.0608	.0387	.0438	.0451
-------	-------	-------	-------	-------

DATA FOR LOT A METHOD 2

LAB A	LAB B	LAB C	LAB D	LAB E
5.9390	5.8360	6.0350	6.1930	5.8630
5.9750	5.7360	5.8300	5.8410	5.9910
5.8930	5.8430	6.0630	5.8480	5.8570
5.9550	5.8250	6.0330	5.9770	5.8490
6.0240	5.9540	5.7610	5.9100	5.9290
5.9600	5.7140	5.8530	5.8020	5.8400
5.8940	5.7030	5.9080	5.9660	5.8940
5.7090	5.7710	5.7570	5.8260	5.8210
5.8900	5.6750	6.0360	5.8730	5.7840
5.9960	5.7630	5.9090	5.7870	5.7030

AVERAGES FOR EACH LAB

5.9235	5.7820	5.9185	5.9023	5.8531
--------	--------	--------	--------	--------

STANDARD DEVIATIONS FOR EACH LAB

.0879	.0837	.1176	.1206	.0782
-------	-------	-------	-------	-------

DATA FOR LOT B METHOD 1

LAB A	LAB B	LAB C	LAB D	LAB E
5.9090	5.9490	5.8820	5.9140	5.9150
5.9470	5.9670	5.8860	5.9800	5.9890
5.9790	5.9260	5.9340	6.0100	5.8360
5.9110	5.9570	5.9180	5.8760	5.8510
5.8700	5.9920	5.9320	5.9070	5.9390
5.9490	5.9600	5.9080	5.9210	5.9090
5.8660	5.9270	5.9570	5.9220	5.9410
5.9100	5.8350	5.8980	5.9010	5.9360
5.9500	5.9250	5.9420	5.9400	5.8980
5.9600	5.9780	5.9430	5.9200	5.8740

AVERAGES FOR EACH LAB

5.9251	5.9416	5.9200	5.9291	5.9088
--------	--------	--------	--------	--------

STANDARD DEVIATIONS FOR EACH LAB

.0381	.0438	.0257	.0391	.0462
-------	-------	-------	-------	-------

DATA FOR LOT B METHOD 2

LAB A	LAB B	LAB C	LAB D	LAB E
6.0990	5.9410	6.1590	6.0940	6.0800
6.1290	6.0390	6.2450	6.0670	5.9900
6.1610	6.0420	5.9940	5.8880	6.0510
6.1120	6.0880	5.9670	6.0050	6.0200
6.0510	5.9000	6.1310	5.9960	6.1260
6.1770	5.8790	6.0090	5.9050	6.1920
5.9640	5.8650	6.0640	5.9370	5.9780
6.2000	5.9120	5.8710	6.0040	5.9830
5.8730	5.8900	6.0940	5.9940	6.0980
6.0370	5.9810	5.9320	6.0020	6.2490

AVERAGES FOR EACH LAB

6.0803	5.9537	6.0466	5.9892	6.0767
--------	--------	--------	--------	--------

STANDARD DEVIATIONS FOR EACH LAB

.1017	.0790	.1136	.0648	.0918
-------	-------	-------	-------	-------

DATA FOR LOT C METHOD 1

LAB A	LAB B	LAB C	LAB D	LAB E
6.0070	5.9920	5.8870	5.9600	6.0110
6.0250	5.9530	6.0100	5.9400	5.9960
5.9840	5.9600	6.1110	5.9910	5.9240
6.0150	5.9030	6.0560	5.9700	6.0340
6.0500	5.9390	6.0160	6.0010	5.8990
6.0180	5.9120	5.9920	5.9760	6.0430
5.9850	5.9260	6.0470	5.9590	5.9979
5.8930	6.0540	5.9950	6.0840	5.9760
5.9830	6.0630	5.9600	5.9510	6.0290
6.0360	6.0510	6.0240	5.9190	5.9900

AVERAGES FOR EACH LAB

5.9996	5.9753	6.0098	5.9751	5.9899
--------	--------	--------	--------	--------

STANDARD DEVIATIONS FOR EACH LAB

.0438	.0611	.0597	.0451	.0467
-------	-------	-------	-------	-------

DATA FOR LOT C METHOD 2

LAB A	LAB B	LAB C	LAB D	LAB E
6.0890	6.1590	6.0040	5.9130	6.2040
6.1450	6.0700	6.1460	6.0040	6.0480
6.0630	6.0170	6.0530	5.9430	6.0860
6.1250	6.2480	5.9420	5.9630	6.2140
6.1940	6.0920	6.0640	6.0720	6.1970
6.1300	6.2770	6.2620	6.0470	6.2880
6.0640	6.1610	6.1380	6.1920	6.0740
5.8790	5.9950	5.9910	6.0360	6.2200
6.1600	6.1980	5.8450	6.0290	5.9970
6.1660	6.3170	6.0550	5.9350	6.0860

AVERAGES FOR EACH LAB

6.1015	6.1534	6.0500	6.0134	6.1414
--------	--------	--------	--------	--------

STANDARD DEVIATIONS FOR EACH LAB

.0894	.1093	.1147	.0823	.0944
-------	-------	-------	-------	-------

APPENDIX D

Results of Analysis of Variance used in Calculating
Repeatability and/or Reproducibility

ANALYSIS OF VARIANCE FOR LOT A METHOD 1

	SS	DF	MS	F
BETWEEN	.006033	4	.001508	.735245
WITHIN	.092315	45	.002051	
TOTAL	.098349	49	.002007	

THE GRAND MEAN FOR LOT A METHOD 1 IS 5.760

ANALYSIS OF VARIANCE FOR LOT A METHOD 2

	SS	DF	MS	F
BETWEEN	.141145	4	.035286	3.583580
WITHIN	.443100	45	.009847	
TOTAL	.584245	49	.011923	

THE GRAND MEAN FOR LOT A METHOD 2 IS 5.876

ANALYSIS OF VARIANCE FOR LOT B METHOD 1

	SS	DF	MS	F
BETWEEN	.005798	4	.001449	.943859
WITHIN	.069106	45	.001536	
TOTAL	.074904	49	.001529	

THE GRAND MEAN FOR LOT B METHOD 1 IS 5.925

ANALYSIS OF VARIANCE FOR LOT B METHOD 2

	SS	DF	MS	F
BETWEEN	.124704	4	.031176	3.701326
WITHIN	.379032	45	.008423	
TOTAL	.503736	49	.010280	

THE GRAND MEAN FOR LOT B METHOD 2 IS 6.029

ANALYSIS OF VARIANCE FOR LOT C METHOD 1

	SS	DF	MS	F
BETWEEN	.009223	4	.002306	.858670
WITHIN	.120835	45	.002685	
TOTAL	.130058	49	.002654	

THE GRAND MEAN FOR LOT C METHOD 1 IS 5.990

APPENDIX E

Calculation of Repeatabilities and Reproducibilities
Shown in Table 6

The following calculations use data taken from the Tables in Appendix D:

Lot A, Method 1

F = .735 is less than F_{.95} (4, 45) = 2.59 therefore the repeatability and reproducibility are the same.

$$s_p^2 = .0020515$$

$$\text{Repeatability} = 2.77 \times ((2.0515 \times 10^{-3})/10)^{.5}$$

$$\text{Repeatability} = .0397$$

Lot B, Method 1

F = .944, Repeatability = Reproducibility

$$s_p^2 = .0015357$$

$$\text{Repeatability} = 2.77 \times ((1.5357 \times 10^{-3})/10)^{.5}$$

$$\text{Repeatability} = .0236$$

Lot C, Method 1

F = .859, Repeatability = Reproducibility

$$s_p^2 = .0026853$$

$$\text{Repeatability} = 2.77 \times (.0026852/10)^{.5}$$

$$\text{Repeatability} = .0454$$

Lot A, Method 2

F = 3.584, Reproducibility ≠ Repeatability

$$s_p^2 = .0098467$$

$$s_m^2 = .0352863$$

$$\text{Repeatability} = 2.77 \times (.0098467/10)^.5$$

$$\text{Repeatability} = .0869$$

$$\sigma_m^2 = \frac{.0352863 - .0098467}{10} = 2.5440 \times 10^{-3}$$

$$\text{Reproducibility} = 2.77 \times (\sigma_m^2 + \sigma^2/10)^.5$$

$$\begin{aligned} \text{Reproducibility} &= 2.77 \times (2.5440 \times 10^{-3} + (9.8467 \times 10^{-3})/10)^.5 \\ &= .1645 \end{aligned}$$

Lot B, Method 2

F = 3.701, Reproducibility \neq Repeatability

$$s_p^2 = .0084229$$

$$s_m^2 = .0804$$

$$\text{Repeatability} = 2.77 \times (.0084229/10)^.5$$

$$\text{Repeatability} = .0804$$

$$\sigma_m^2 = \frac{.031176 - .0084229}{10}$$

$$\sigma_m^2 = 2.275 \times 10^{-3}$$

$$\begin{aligned} \text{Reproducibility} &= 2.77 \times (\sigma_m^2 + \sigma^2/10)^.5 \\ &= 2.77 \times (2.275 \times 10^{-3} + .0084229/10)^.5 \\ &= .1547 \end{aligned}$$

APPENDIX F

Computer Programs used in Evaluating ANOVA

```

720 107 FORMAT (IX, XIER = 3, I3)
730 C
740 STOP
750 END
---
```

```

PROGRAM HAROLD(INPUT, OUTPUT, TAPES-INPUT, TAPES-OUTPUT) 740

```

```

THIS IS THE PROGRAM WHICH CALLS THE INSL SUBROUTINE
ACRDN TO PERFORM A ONE WAY ANALYSIS OF VARIANCE ON
THE GIVEN DATA. THIS PARTICULAR DATA SET IS THE DATA
FOR LOT C METHOD 2.

```

```

DIMENSION VEX(3)
DIMENSION VEX(3)
INTEGER NT, NUS, NDF(3), IER
REAL Y(50), TH(5), UTU(5), S(3), CM
DATA VEX / 2HT, 3HTU, 1HS /
DATA Y/6.089, 6.145, 6.063, 6.125, 6.104,
+6.130, 6.064, 5.879, 6.160, 6.166,
+6.159, 6.070, 6.017, 6.248, 6.092,
+6.277, 6.161, 5.995, 6.198, 6.317,
+6.004, 6.146, 6.053, 5.942, 6.064,
+6.262, 6.138, 5.991, 5.845, 6.055,
+5.913, 6.004, 5.943, 5.963, 6.072,
+5.047, 6.102, 6.036, 6.020, 6.035,
+5.284, 6.048, 6.086, 6.214, 6.197,
+6.288, 6.074, 6.220, 5.997, 6.086 /
DATA N/10, 10, 10, 10, 10 /
NT=5

```

```

CALL CONNOC(SLINPUT)
CALL CONNOC(OUTPUT)
CALL ACRDN (Y, NT, N, TH, UTU, S, CM, NDF, IER)
VEX(1)=S(1)/NDF(1)
VEX(2)=S(2)/NDF(2)
VEX(3)=S(3)/NDF(3)
FRATIO=VEX(1)/VEX(2)
PRINT 108, FRATIO
108 FORMAT (IX, XFR=3, F10.3)
PRINT 102, MS, VEX(1), VEX(2), VEX(3)
PRINT 101, VEX(1), TH
PRINT 101, VEX(2), UTU
PRINT 100, VEX(3), S
109 FORMAT (IX, A2, X = (3, 3F10.5, 3)X )
101 FORMAT (IX, A2, X = (3, 5F10.5, 3)X )
102 FORMAT (IX, A2, RMS = (3, 3F12.7, 3)X )
PRINT 105, CM
105 FORMAT (IX, XCM=3, F10.3)
PRINT 103, NDF
103 FORMAT (IX, XNDF = (3, 2(I2, 3, 3), 12, 3)X )
PRINT 107, IER

```



```

100 C
110 C
120 C
130 C
140 C
150 C
160 C
170 C
180 C
190 C
200 C
210
220
230
240 C
250
260
270
280
290
300
310
320
330
340
350 C
360
370
380
390
400
410
420 C
430
440
---
```

PROGRAM HAROLD(INPUT, OUTPUT, TAPES-INPUT, TAPES-OUTPUT)

THIS IS A PROGRAM USED IN EVALUATING TERMS IN THE ONE WAY ANALYSIS
OF VARIANCE COMPUTATION. IT COMPUTES THE SQUARE OF EACH DATA POINT
AND THE SUM OF THE SQUARES OF ALL THE DATA POINTS.

```

      DIMENSION Y(50)
      X = 0.
      OLD = 0.

      DATA Y/6.089,6.145,6.063,6.125,6.194,
+6.130,6.064,5.879,6.160,6.166,
+6.159,6.070,6.017,6.248,6.092,
+6.277,6.161,5.995,6.198,6.317,
+6.004,6.146,6.053,5.942,6.064,
+6.262,6.138,5.991,5.845,6.055,
+5.913,6.004,5.943,5.963,6.072,
+6.047,6.192,6.036,6.029,5.935,
+6.204,6.048,6.086,6.214,6.197,
+6.288,6.074,6.220,5.997,6.086/

      DO 10, I=1,50
      X = Y(I)*X*2
      OLD = OLD + X
      PRINT 100, X, OLD
      100 FORMAT (2F 20.5)
      10 CONTINUE

      STOP
      END

```

```

100 C
110 C
1101 C
1102 C
1103 C
1104 C
1105 C
1106 C
1107 C
1108 C
1109 C
1110 C
1111 C
1112 C
1113 C
1114 C
1115 C
1116 C
1117 C
1118 C
1119 C
1120 C
1121 C
1122 C
1123 C
1124 C
1125 C
1126 C
1127 C
1128 C
1129 C
1130 C
1131 C
1132 C
1133 C
1134 C
1135 C
1136 C
1137 C
1138 C
1139 C
1140 C
1141 C
1142 C
1143 C
1144 C
1145 C
1146 C
1147 C
1148 C
1149 C
1150 C
1151 C
1152 C
1153 C
1154 C
1155 C
1156 C
1157 C
1158 C
1159 C
1160 C
1161 C
1162 C
1163 C
1164 C
1165 C
1166 C
1167 C
1168 C
1169 C
1170 C
1171 C
1172 C
1173 C
1174 C
1175 C
1176 C
1177 C
1178 C
1179 C
1180 C
1181 C
1182 C
1183 C
1184 C
1185 C
1186 C
1187 C
1188 C
1189 C
1190 C
1191 C
1192 C
1193 C
1194 C
1195 C
1196 C
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PROGRAM ERIC(INPUT, OUTPUT, TAPE3, TAPES-INPUT, TAPES-OUTPUT)
 THIS IS THE PROGRAM WHICH CALLS THE INSL SUBROUTINE ARCBAN
 IN ORDER TO EVALUATE THE DATA FOR EACH METHOD USING A TWO-
 WAY CLASSIFICATION ANALYSIS OF VARIANCE TECHNIQUE.
 REAL V(150), EN(23), CM, S(5)
 READ (3, 25) Y
 25 FORMAT (15X, F5.4, 3X, F5.4, 3X, F5.4, 3X, F5.4, 3X, F5.4)
 CALL CONNEX(SLINPUT)
 CALL CONNEX(SLOUTPUT)
 MR=10
 NB=3
 NT=5
 CALL ARCBAN (V, MR, NB, NT, EM, CM, S, NDF, IER)
 PRINT 100, EM
 100 FORMAT (11F7.3, //, 12F7.3, ///)
 PRINT 101, CM
 101 FORMAT (F5.2)
 PRINT 102, S
 102 FORMAT (5F10.4)
 PRINT 103, NDF
 103 FORMAT (I5)
 PRINT 104, IER
 104 FORMAT (I2)
 STOP
 END

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PROGRAM ERIC(INPUT, OUTPUT,TAPE3, TAPES-INPUT, TAPES-OUTPUT)

THIS PROGRAM PRINTS OUT THE TWO WAY CLASSIFICATION DATA

DIMENSION Y(150),Z(50),X(50)

HEAD (3,25) Y

25 FORMAT(15X,F5.3,X,F5.3,X,F5.3,X,F5.3,X,F5.3,X,F5.3)

PRINT 105

105 FORMAT(27X,1TWO WAY CLASSIFICATION-DATA OF METHOD 12,////////)

101 FORMAT(24X,SLAB A2,9X,SLAB B2,9X,SLAB C2,9X,SLAB D2,9X,SLAB E2)

PRINT 120

120 FORM (//)

PRINT 100, Y(1), Y(11), Y(21), Y(31), Y(41)

PRINT 100, Y(2), Y(12), Y(22), Y(32), Y(42)

PRINT 100, Y(3), Y(13), Y(23), Y(33), Y(43)

PRINT 100, Y(4), Y(14), Y(24), Y(34), Y(44)

PRINT 100, Y(5), Y(15), Y(25), Y(35), Y(45)

PRINT 100, Y(6), Y(16), Y(26), Y(36), Y(46)

PRINT 100, Y(7), Y(17), Y(27), Y(37), Y(47)

PRINT 100, Y(8), Y(18), Y(28), Y(38), Y(48)

PRINT 100, Y(9), Y(19), Y(29), Y(39), Y(49)

PRINT 110, Y(10), Y(20), Y(30), Y(40), Y(50)

110 FORMAT (15X,5F14.4,////)

100 FORMAT (15X,5F14.4)

90 FORMAT (5X,10T 02,5X,5F14.4)

DO 20 I=51,100

J=1-50

Z(J) = Y(I)

30 CONTINUE

PRINT 200, Z(1), Z(11), Z(21), Z(31), Z(41)

PRINT 200, Z(2), Z(12), Z(22), Z(32), Z(42)

PRINT 200, Z(3), Z(13), Z(23), Z(33), Z(43)

PRINT 200, Z(4), Z(14), Z(24), Z(34), Z(44)

PRINT 200, Z(5), Z(15), Z(25), Z(35), Z(45)

PRINT 200, Z(6), Z(16), Z(26), Z(36), Z(46)

PRINT 200, Z(7), Z(17), Z(27), Z(37), Z(47)

PRINT 200, Z(8), Z(18), Z(28), Z(38), Z(48)

PRINT 200, Z(9), Z(19), Z(29), Z(39), Z(49)

PRINT 210, Z(10), Z(20), Z(30), Z(40), Z(50)

210 FORMAT (15X,5F14.4,////)

200 FORMAT (15X,5F14.4)

200 FORMAT (5X,10T 02,5X,5F14.4)

DO 20 I=101,150

J=1-100

X(J) = Y(I)

20 CONTINUE

PRINT 300, X(1), X(11), X(21), X(31), X(41)

PRINT 300, X(2), X(12), X(22), X(32), X(42)

PRINT 300, X(3), X(13), X(23), X(33), X(43)

PRINT 300, X(4), X(14), X(24), X(34), X(44)

PRINT 300, X(5), X(15), X(25), X(35), X(45)

PRINT 300, X(6), X(16), X(26), X(36), X(46)

PRINT 300, X(7), X(17), X(27), X(37), X(47)

PRINT 300, X(8), X(18), X(28), X(38), X(48)

PRINT 300, X(9), X(19), X(29), X(39), X(49)

PRINT 310, X(10), X(20), X(30), X(40), X(50)

310 FORMAT (15X,5F14.4,////)

300 FORMAT (15X,5F14.4)

100 FORMAT (5X,10T 02,5X,5F14.4)

STOP

END

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PROGRAM ERIC(INPUT,OUTPUT,TAPE3,TAPE5-INPUT,TAPE5-OUTPUT) C
DO 5000 I=1,15
U=U+SUM(I)
CONTINUE
PRINT 2000,SUM(1),SUM(2),SUM(3),SUM(4),SUM(5),ROW(1)
PRINT 2000,SUM(6),SUM(7),SUM(8),SUM(9),SUM(10),ROW(2)
PRINT 2000,SUM(11),SUM(12),SUM(13),SUM(14),SUM(15),ROW(3)
PRINT 2000,COL(1),COL(2),COL(3),COL(4),COL(5),M
FORMAT(15X,F10.4,3X,F10.4,////) C
STOP
END

THIS IS THE PROGRAM WHICH PRINTS OUT THE TOTALS
AND SUBTOTALS FOR USE IN THE TWO-WAY CLASSIFICATION
ANALYSIS OF VARIANCE CALCULATIONS.

DIMENSION Y(150),SUM(15),ROW(3),COL(5),SOROW(3),SECOL(5),750
READ (3,25) Y
25 FORMAT (15X,F5.3,3X,F5.3,3X,F5.3,3X,F5.3)
SUM = 0.
O=0.
C=0.
R=0.
SUB=0.
UP=0.
X=0.
U=0.
Z=0.
PRINT 105
105 FORMAT(30X,3 DATA OF METHOD 1-TOTALS AND SUBTOTALS,////)
PRINT 101
101 FORMAT(20X,SLAB A2,5X,SLAB B2,5X,SLAB C2,5X,SLAB D2,5X,SLAB E2)
PRINT 120
120 FORMAT (//)
DO 35 J=1,15
K=10*(J-1)+1
L=K+9
DO 40 M=K,L
SUM = V(M) + SUM
SUM(J) = SUM
CONTINUE
SUM = 0.
35 CONTINUE
DO 800 I=1,3
K=5*(I-1)+1
L=K+4
DO 900 M=K,L
R = R + SUM(M)
ROW(I)=R
CONTINUE
R=0.
900 CONTINUE
800 CONTINUE
DO 1100 I=1,5
K=1
L=K+10
DO 1200 M=K,L,5
C=C+SUM(M)
COL(I)=C
CONTINUE
1200 CONTINUE

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PROGRAM ERIC(INPUT, OUTPUT, TAPE3, TAPES-INPUT, TAPES-OUTPUT)

THIS IS THE PROGRAM WHICH CALLS THE IMSL SUBROUTINE
ARC4BN AND PRINTS OUT THE ANALYSIS OF VARIANCE
RESULTS FOR A TWO WAY CLASSIFICATION OF THE DATA FOR
METHOD 1.

INTEGER NR(10),NB(3),NT(5),NDF(5),IER
REAL Y(150),EM(23),GM,S(5),MS(5),F(3)

READ (3,25) Y
25 FORMAT (15X,F5.4,3X,F5.4,3X,F5.4,3X,F5.4,3X,F5.4)

CALL CONNCT(SLINPUT)
CALL CONNCT(OUTPUT)

NP=10
NB=3
NT=5
CALL ARC4BN (Y,NR,NB,NT,EM,GM,S,NDF,IER)

DO 10 I=1,5
MS(I)=S(I)/NDF(I)
10 CONTINUE

F(1)=MS(1)/MS(3)
F(2)=MS(2)/MS(3)
F(3)=MS(3)/MS(4)

PRINT 2000
2000 FORMAT (23X,'TWO WAY ANOVA-METHOD 1',////)

PRINT 1000
1000 FORMAT (23X,'SS',15X,'Df',12X,'MS',6X,'F-RATIO')
PRINT 500
500 FORMAT (////)

PRINT 102, S(1),NDF(1),MS(1),F(1)
102 FORMAT (5X,'SOURCE',10X,F10.4,8X,15.8X,F7.4,3X,F9.4)

PRINT 103,S(2),NDF(2),MS(2),F(2)
103 FORMAT (5X,'COLUMN',7X,F10.4,8X,15.8X,F7.4,3X,F9.4)

PRINT 104, S(3),NDF(3),MS(3),F(3)
104 FORMAT (5X,'INTERACTION',2X,F10.4,8X,15.8X,F7.4,3X,F9.4)

PRINT 105, S(4),NDF(4),MS(4)
105 FORMAT (5X,'WITHIN',8X,F10.4,8X,15.8X,F7.4)

PRINT 106, S(5),NDF(5)
106 FORMAT (5X,'TOTAL',8X,F10.4,8X,15)

PRINT 3000
3000 FORMAT (//////////)

STOP
END

TWO WAY CLASSIFICATION-DATA OF METHOD 2

	LAB A	LAB B	LAB C	LAB D	LAB E
LOT A	5.9390	5.8360	6.0350	6.1930	5.8630
	5.9750	5.7360	5.8300	5.8410	5.9910
	5.8330	5.8430	6.0630	5.8430	5.8570
	5.9550	5.8250	6.0330	5.9770	5.8490
	6.0240	5.9540	5.7610	5.9100	5.9230
	5.9690	5.7140	5.8530	5.8050	5.8400
	5.8940	5.7030	5.9080	5.9660	5.8940
	5.7090	5.7710	5.7570	5.8260	5.8210
	5.8900	5.6750	6.0360	5.8730	5.7840
	5.9960	5.7630	5.9090	5.7870	5.7030
LOT B	6.0990	5.9410	6.1590	6.0940	6.0800
	6.1290	6.0390	6.2450	6.0670	5.9900
	6.1610	6.0420	5.9940	5.8880	6.0510
	6.1120	6.0880	5.9670	6.0950	6.0200
	6.0510	5.9000	6.1310	5.9960	6.1260
	6.1770	5.8790	6.0090	5.9050	6.1920
	5.9640	5.8650	6.0640	5.9370	5.9780
	6.2080	5.9120	5.8710	6.0040	5.9830
	5.8730	5.8900	6.0940	5.9940	6.0980
	6.0370	5.9810	5.9320	6.0020	6.2490
LOT C	6.0990	6.1590	6.0040	5.9130	6.2040
	6.1450	6.0700	6.1460	6.0040	6.0480
	6.0630	6.0170	6.0530	5.9430	6.0860
	6.1850	6.2480	5.9420	5.9630	6.2140
	6.1940	6.0920	6.0640	6.0720	6.1970
	6.1360	6.2770	6.2620	6.0470	6.2890
	6.0640	6.1610	6.1380	6.1920	6.0740
	5.8700	5.9950	5.9910	6.0360	5.9300
	6.1600	6.1980	5.8450	6.0250	5.9970
	6.1660	6.3170	6.0550	5.9350	6.0660

DATA OF METHOD 2-TOTALS AND SUBTOTALS

LAB A	LAB B	LAB C	LAB D	LAB E	
59.2350	57.8200	59.1850	59.0230	58.5310	293.7940
60.8030	59.5370	60.4660	59.8920	60.7670	301.4650
61.0150	61.5340	60.5000	60.1340	61.4140	304.5970
181.0530	178.8910	180.1510	179.0490	180.7120	899.8560

[illegible]

[illegible]

```

PROGRAM HAROLD INPUT, OUTPUT, TAPES INPUT, TAPES OUTPUT)
      LTBH= 15:04 PM
      DATA FOR LOT B METHOD 1
      PR= .94
      RPS = (.0014435, .001357, .0015861,
      RM = (.5, 2.0, 5.0, 10.0, 20.0, 50.0, 100.0,
      ST = (.00145, .0012, .0015,
      S = (.00180, .0011, .0015),
      GMF = (.5, 2.0, 5.0, 10.0, 20.0, 50.0, 100.0,
      IER = (.4, 5.0, 10.0)
      END
      *****
      410000 CM STORAGE USED
      5.336 CP SECONDS COMPIATION TIME
      0.1700 MAXIMUM EXECUTION PL
      .1210 CP SECONDS EXECUTION TIME.

```


APPENDIX G

User's Guide for this Document

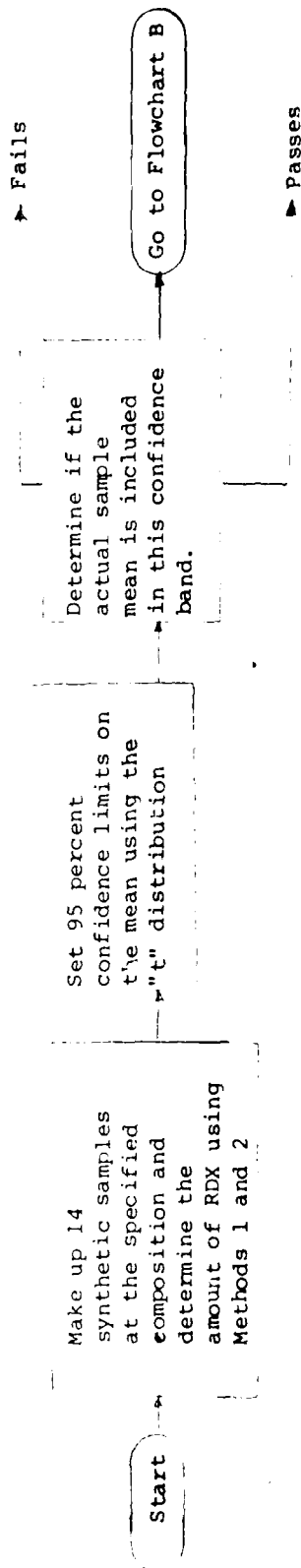
The following flow charts are a schematic representation of the operations (chemical analysis and data collection) and statistical procedures used in this text.

The flow charts A, B, and C correspond to Parts 1 and 2 of the Statistical Guide. The proponent of the new test method should do all the tests and procedures shown in these flow charts. The results of these tests provide information as to the accuracy and precision of the new test method as compared to the old test method. These results form the basis for making a decision as to whether or not a full scale round robin should be conducted.

Flow charts D and E (corresponding to Parts 3 and 4) show the operations involved in evaluating the round robin data in the event tha the decision is made to conduct the round robin. The operations involved in Parts 3 and 4 are basically techniques to evaluate the method repeatability and reproducibility. At first glance, the reader may not be able to distinguish the difference between these two parts. The difference has to do with the degrees of freedom used in the repeatability and reproducibility estimates. In part 3 there are three estimates of repeatability and/or reproducibility for each method based on 45 degrees of freedom. Each estimate comes from the data for a given lot. In part 4 a single estimate of the repeatability and/or reproducibility with 135 degrees of freedom is obtained for each method. This single estimate is based on the combined data for all three lots.

FLOW CHART A: EVALUATION OF ACCURACY OF EACH METHOD

Pages 1 - 3 in Part 1



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STATISTICAL GUIDE FOR DETERMINING THE EQUIVALENCY OF TWO CHEMIC--ETC(U)
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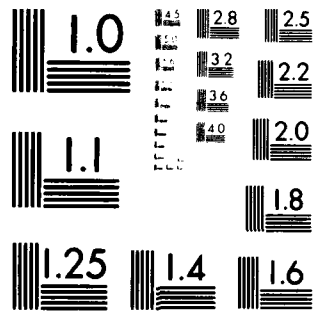
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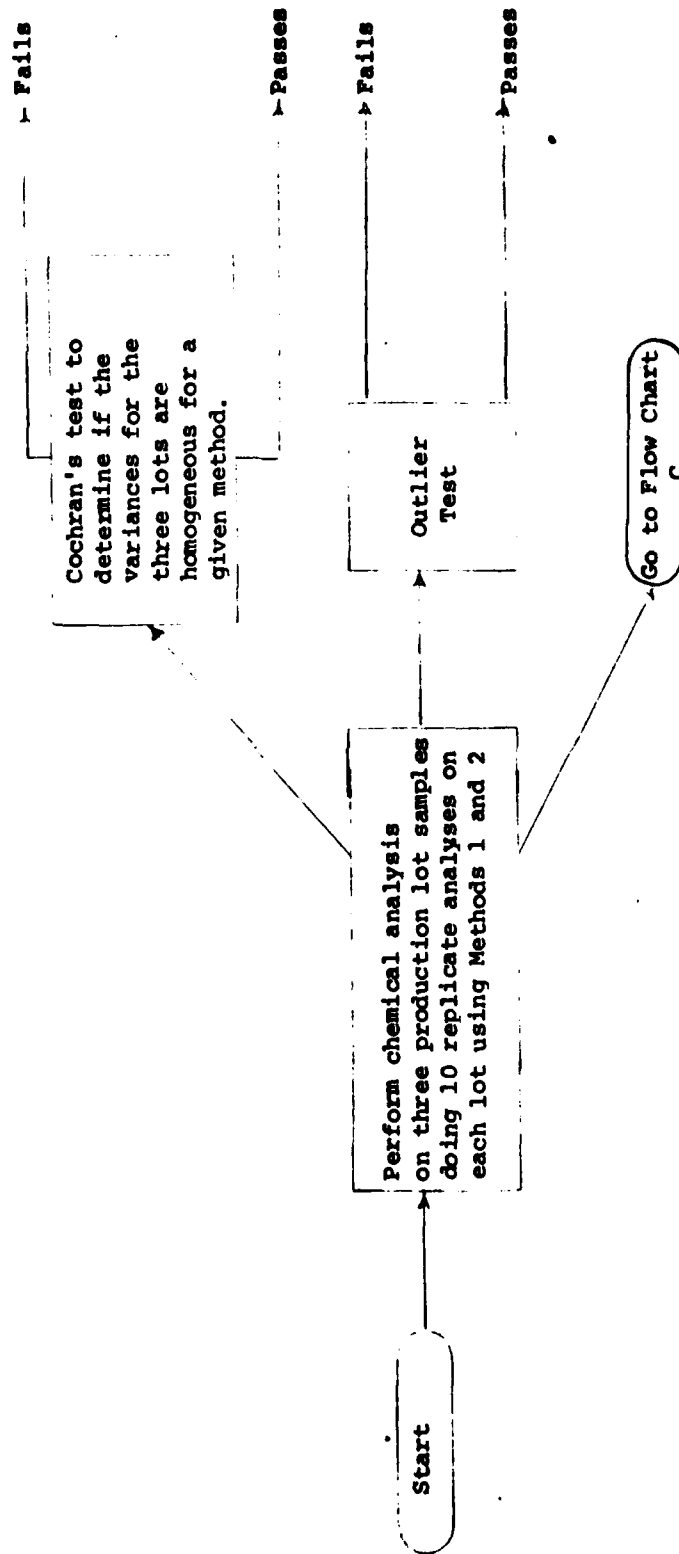
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MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS 1963-A

FLOW CHART B: EVALUATION OF PRODUCTION LOT AND/OR TEST METHOD VARIABILITY

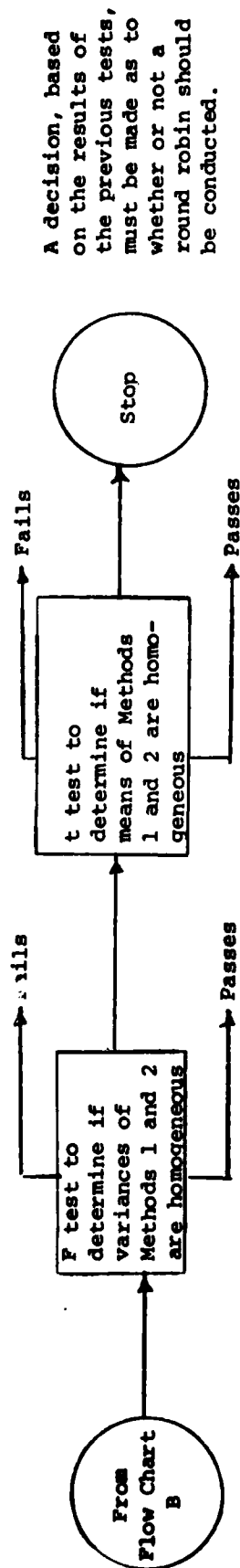
Pages 7 - 10 in Part 2



Conclusions: If a test method fails to exhibit homogeneity of variances for three lots or shows outliers in the data, either the lot variability or the test method may be suspect.

FLOW CHART C: CONTRAST OF MEANS AND VARIANCES OF METHODS

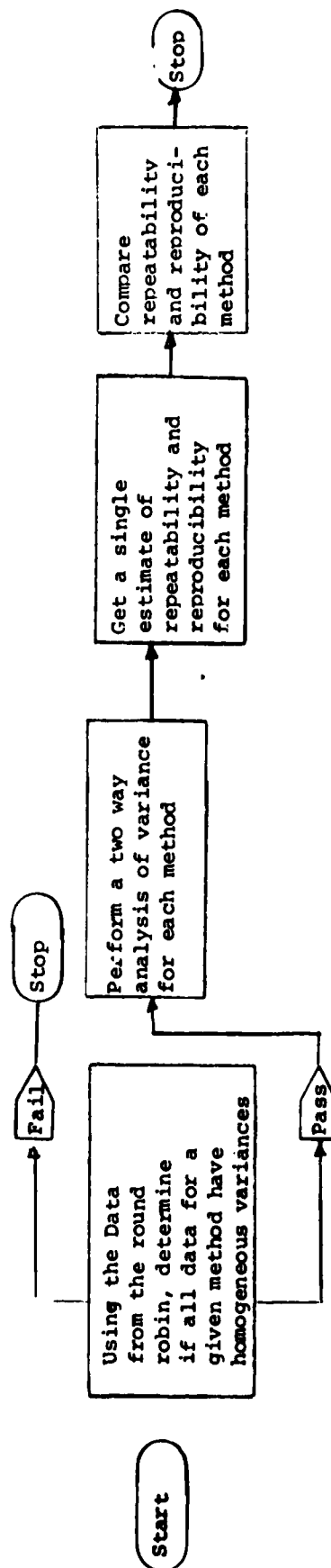
Pages 11 - 13 in Part 2



Conclusions: If the two test methods do not exhibit homogeneous means and variances, they are not equivalent.

FLOW CHART E: A SINGLE ESTIMATE OF REPEATABILITY AND REPRODUCIBILITY FOR EACH METHOD

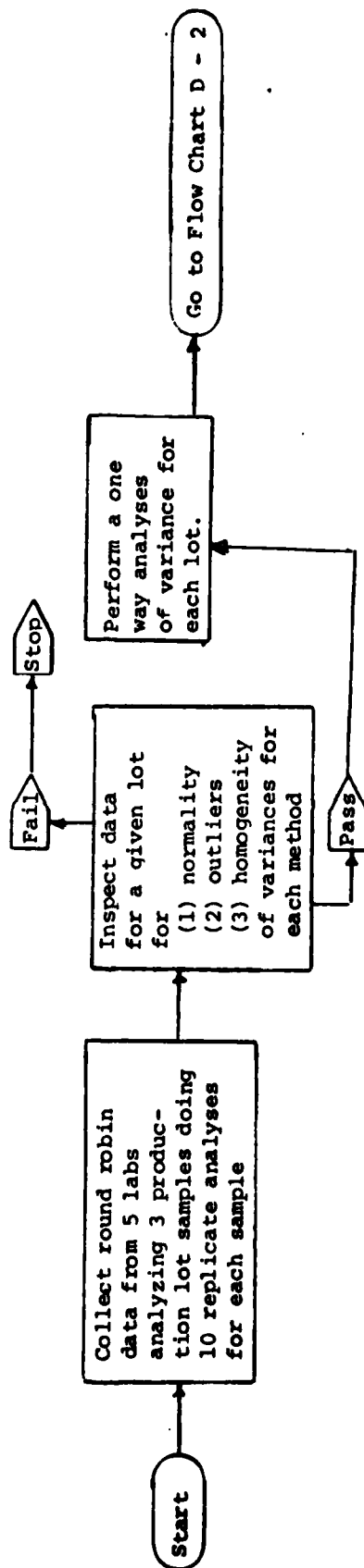
Pages 26 - 39 in Part 2



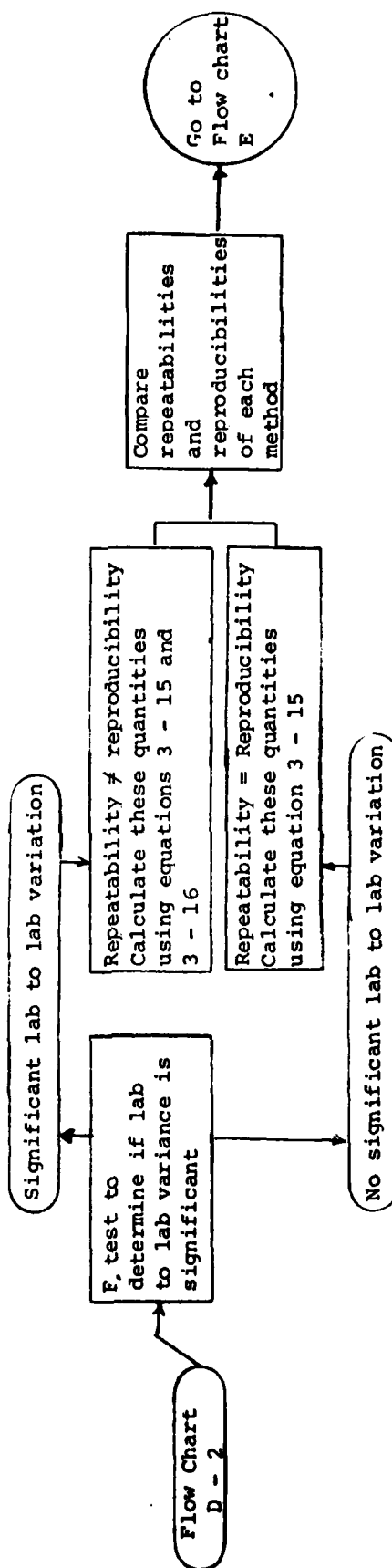
Conclusions: Both methods must have the same repeatability and reproducibility to be equivalent

FLOW CHART D - 1: ESTIMATES OF REPEATABILITY AND REPRODUCIBILITY
FOR EACH LOT X METHOD COMBINATION
FROM ROUND ROBIN DATA

Pages 15 - 25 in Part 3



FLOW CHART D - 2: ESTIMATES OF REPEATABILITY AND REPRODUCIBILITY
FOR EACH LOT X METHOD COMBINATION
FROM ROUND ROBIN DATA



Conclusions: Both methods must have the same repeatabilities and reproducibilities to be equivalent

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